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NUARY 3, 1944
OLUME 33, No. 1

AMERICAN DYESTUFF REPORTER

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of the
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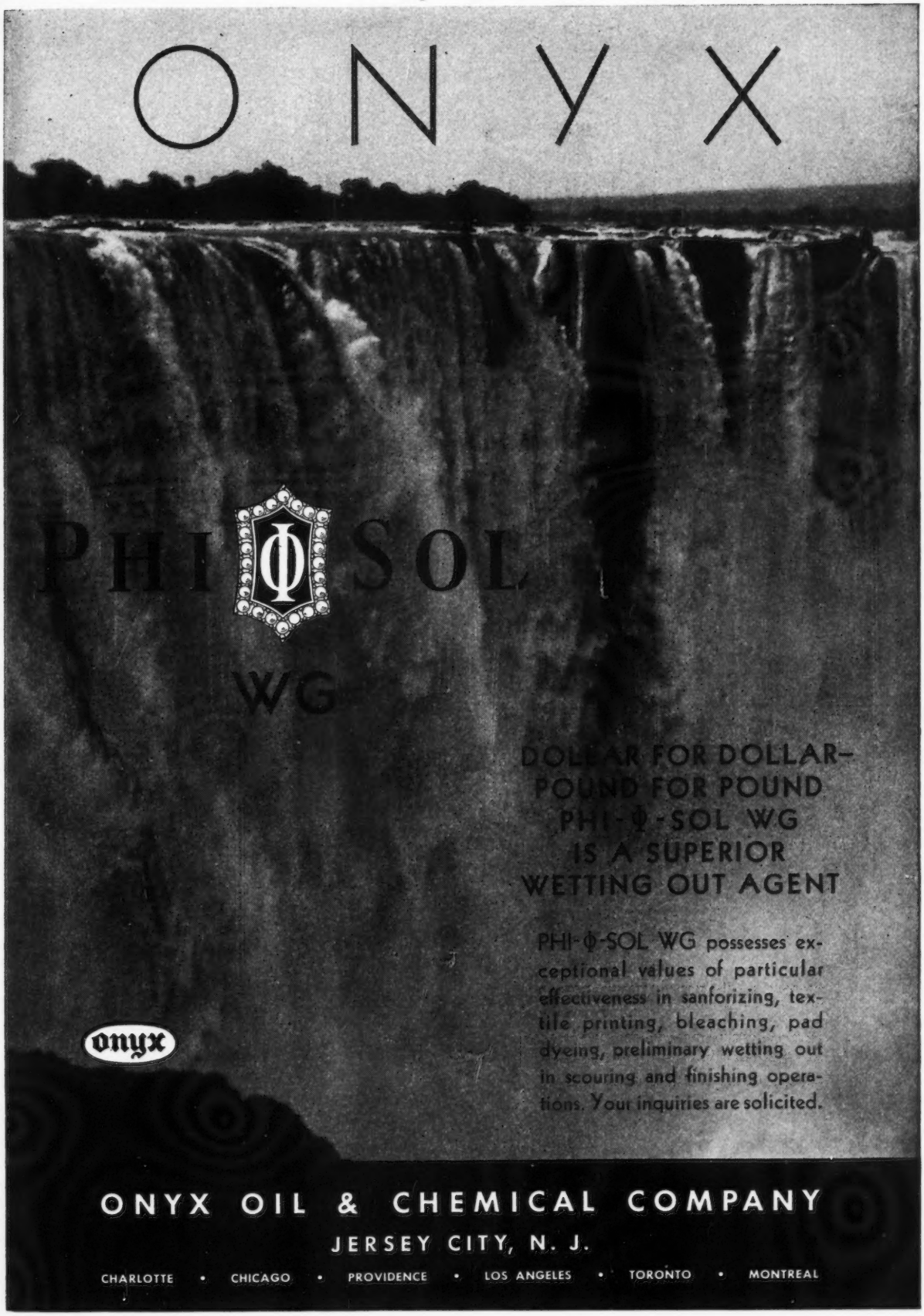
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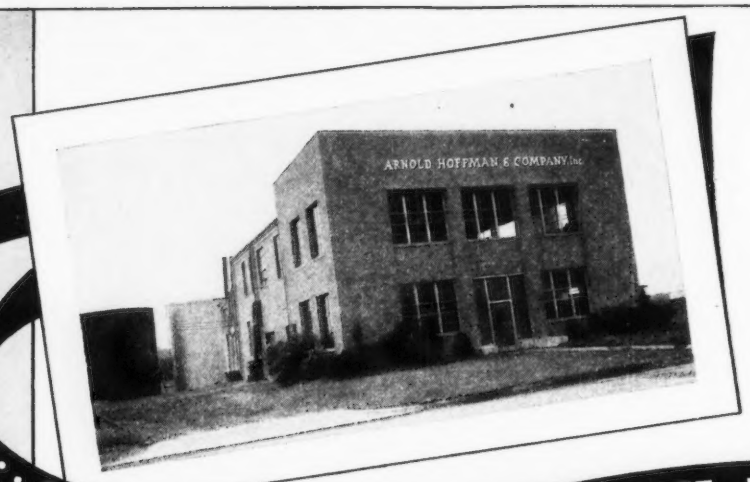

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Concentrated Cotton Warp Size

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There are no boundaries . . . no limits to this world war. ★ It swings from the bleak wastes of the Aleutians to the fore-front of the European fortress . . . from ice-gripped Greenland to war scarred China. In this fierce conflict . . . there is no giving quarter no going half way . . . it is all-out all down the line. Our armed forces, valient in giving their best . . . deserve the best we can give them. ★ By far sighted planning General continues to follow through on all government orders . . . placing its strategic materials in thousands of vital places with the studied timing of an artillery barrage.

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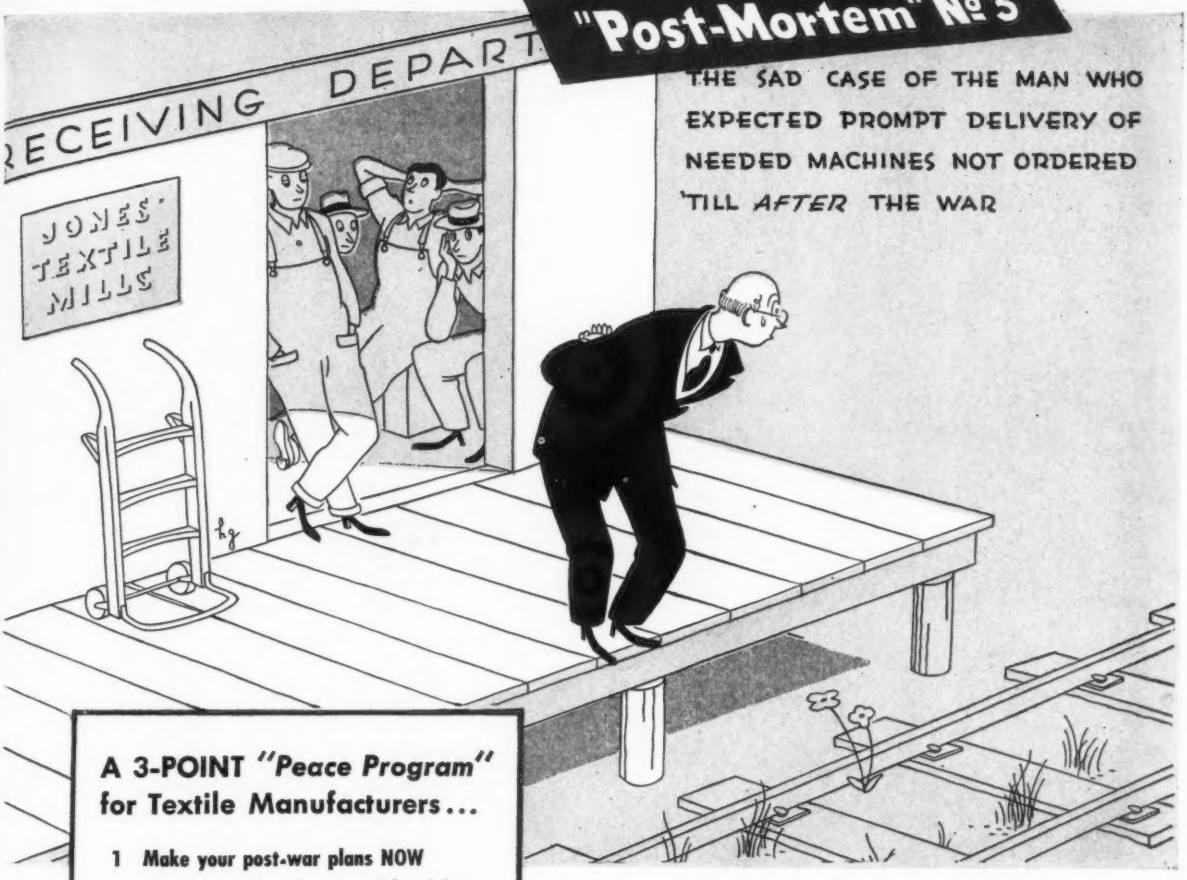
POST-WAR "Post-Mortem" No. 3

THE SAD CASE OF THE MAN WHO EXPECTED PROMPT DELIVERY OF NEEDED MACHINES NOT ORDERED 'TILL *AFTER* THE WAR



A 3-POINT *"Peace Program"* for Textile Manufacturers...

1 **Make your post-war plans NOW**
don't let peace catch you without definite plans for products, markets, sales and production.

2 **Decide on new equipment needs NOW**
don't wait until the war is over before making a thorough survey of your production equipment and deciding what machines must be replaced.

3 **Order your new equipment NOW**
don't risk serious delay in securing necessary new equipment for post-war production. Metals and other basic materials will continue to be scarce for a long time after the war ends.

If you are going to need new dyeing and finishing equipment after the war, we earnestly recommend that you place your order with us *now*. Our "Post-war Purchase Plan" assures you prompt delivery when war restrictions are lifted. Write *now* for complete details of this plan.

The textile manufacturer who waits until the war is over to order needed new machines, very probably will find himself in an awkward situation, to say the least. The huge backlog of demand for new machines, dammed up by wartime restrictions, will break loose like a flood. Machinery manufacturers, still faced with continuing metal shortages, will be unable to meet *all* these requirements promptly. Result? A long wait for equipment by those who delayed their orders.

But the textile manufacturer who takes time now to *anticipate* his post-war requirements and place his orders, will be in the driver's seat. Without any interruption to your current war production, you should take stock of your future replacement requirements for dyeing and finishing machines. Then place your orders *now* for prompt delivery as soon as government restrictions are lifted.

Ask us about our "Post-War Purchase Plan"

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"Why shouldn't I buy it? I've got the money!"

Sure you've got the money. So have lots of us. And yesterday it was all ours, to spend as we darn well pleased. But not today. Today it isn't ours alone.

"What do you mean, it isn't mine?"

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"You think I can really keep prices down?"

If you don't, who will? Uncle Sam can't do it alone. Every time you refuse to buy something you don't need, every time you refuse to pay more than the ceiling price, every time you shun a black market, you're helping to keep prices down.

"But I thought the government put a ceiling on prices."

You're right, a price ceiling for your protection. And it's up to you to pay no more than the ceiling price. If you do, you're party to a black market deal. And black markets not only boost prices—they cause shortages.

"Doesn't rationing take care of shortages?"

Your ration coupons will—if you use them wisely. Don't spend them unless you have to. Your ration book merely sets a limit on your purchases. Every coupon you don't use today means that much more for you—and everybody else—to share tomorrow.

"Then what do you want me to do with my money?"

Save it! Put it in the bank! Put it in life insurance! Pay off old debts and don't make new ones. Buy and hold War Bonds. Then your money can't force prices up. But it can speed the winning of the war. It can build a prosperous nation for you, your children, and our soldiers, who deserve a stable America to come home to. Keep your dollars out of circulation and they'll keep prices down. The government is helping—with taxes.

"Now wait! How do taxes help keep prices down?"

We've got to pay for this war sooner or later. It's easier and cheaper to pay as we go. And it's better to pay more taxes NOW—while we've got the extra money to do it. Every dollar put into taxes means a dollar less to boost prices. So . . .

Use it up . . . Wear it out . . .
Make it do . . . Or do without

HELP US KEEP PRICES DOWN

A United States war message prepared by the War Advertising Council, approved by the Office of War Information, and contributed by the Magazine Publishers of America

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See those stockings . . . they may be rayon, cotton, or lisle, but if they're not made to last . . . if they don't have that proper soft, clear stitch and smoky, eye-caressing dullness . . . they're "out"!

Yes, it's no news that these days with women more exacting and materials more varying than ever before, improper finishing can break even the strongest brand name.

For months our staff has been working "'round-the-clock" to develop formulae for handling all types of problem-materials. That's why a talk with one of our field men, can help you, as it has helped others, to make "lasting" hose, finished the Fanco way.

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FUNGICIDE M: a single product combining a mildew inhibitor and water repellent for producing a mildewproof water repellent finish on insect netting, tapes, sheeting, webbing, thread, etc., in one operation.

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FUNGICIDE GM: used in conjunction with water repellents in a one bath treatment to mildewproof cottons and impart a water repellent finish.

FUNGICIDE A: a mildewproofer for cloth coating processes . . . — used directly in the vinyl resin solution.

AQUAROL*: produces a water repellent finish on uniform cloth, overcoatings and other military fabrics . . . — may be used in a single bath treatment with Fungicide GM to impart water repellency and mildew resistance to cottons . . . — also used in single bath with Arko Fire Retardant to produce water repellency and fire resistance.

ARKO FIRE RETARDANT: for flameproofing uniform cloth, tentage, felts, etc.

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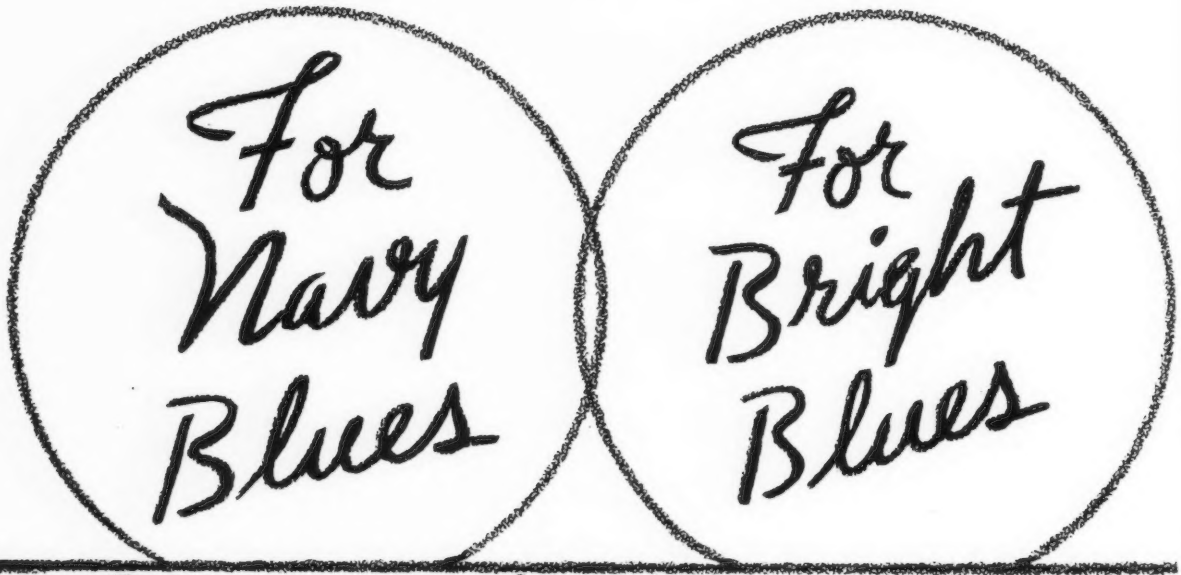

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AMERICAN DYESTUFF REPORTER

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JANUARY 3, 1944

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DYESTUFF REPORTER

VOLUME 33 JANUARY 3, 1944 NUMBER 1

SPECTROPHOTOMETRY AND THE COLORIST

E. I. STEARNS

(A contribution from the Research Department, Calco Chemical Division, American Cyanamid Co.)

SUMMARY

It is pointed out how the colorist, using the spectrophotometer as a tool to assist him, can gain additional information on his problems. The two aspects of interpretation of spectrophotometric data, colorimetry and analysis, are discussed. Applications of these methods of interpretation to twelve mill production and research problems are suggested.

SPECTROPHOTOMETERS have been used by scientists for a number of years. The early types were often visual, that is, the observer had to compare two lights and make adjustments until they were equal to get a single point. They were also non-recording and these single points had to be plotted by hand to get a curve. Because they were so slow they did not find much use in industry.

In the last ten years two instruments have been developed which produce curves of color very rapidly. These are the General Electric automatically recording visual range spectrophotometer[1] (referred to in this paper simply as VS for brevity) and the Snow spectrophotometer (discussed by Monego and von Bergen).[26] These two instruments, and in particular the General Electric type, have found an ever-increasing use in the textile industry. It seems appropriate to review at this time the fields of usefulness of these machines.

The VS (spectrophotometric) plot is a curve of color. White light consists of a mixture of all the colors of the rainbow as can be demonstrated with a triangular piece of glass known as a prism. A colored object is colored because it selectively reflects some of the spectral colors to a greater extent than the others. Thus a blue piece of cloth appears blue to the normal eye because it reflects the blue portion of white light to the eye but absorbs the other portions of the white light.

To describe light reflected from a colored object in a physical sense it is necessary to measure how much of each kind of light is reflected. This is the measurement that the VS makes. In the VS white light is first separated into its rainbow colors by the prisms that comprise the "spectro" part. These rainbow colors are described in terms of millimicrons and are plotted along the bottom of the graph paper. Each of the spectral colors is in turn divided into two beams of light, one of which is directed onto a white standard while the other shines on the sample. The "photometer" part of the VS then measures the ratio of amount of light reflected by the sample to the amount reflected by the white. This ratio is plotted on the graph paper up the side and is commonly expressed in per cent.

Any VS reflectance curve is merely a plot of amount of reflected light versus kind of light and is of itself of little interest. It is the interpretation of the curve, and the inferences which may be drawn that are of interest. In general there are three methods of interpretation. These may be illustrated by considering the VS curve of a piece of wool dyed with Orange II (CI 151). First, only the particular atoms and atomic arrangement found in the dye molecule of Orange II will reflect light in just that way. Hence it may be possible to deduce the chemical structure from the curve. Work of this type has been done by many scientists, notably W. R. Brode, but has not been worked out in detail. This interpretation will not be cosidered in this article. Second, all normal observers looking at the dyeing would agree that it was orange. Hence it should be possible to deduce from the curve alone that the sample will appear visually orange. This is known as a colorimetric interpretation. Third, every time a 1 per cent dyeing of Orange II on wool is made under similar conditions a similar sample and hence a similar VS curve will result. Hence it should be possible to deduce from the curve alone what percentage of what dye was used. This is called analytical interpretation.

It is the colorimetric and analytical interpretation that find the greatest application in the textile industry. Accordingly, in the following discussion, the subject of colorimetry is first reviewed, and then the subject of quantitative analysis. Having an understanding of these two methods of interpretation, the various mill problems are reviewed. To answer a particular question, one or both methods of interpretation may be used to advantage.

COLORIMETRY

Having a VS curve, it is possible to give a description of the color. This is colorimetry.[2] As is well known by color matchers, using a red, yellow and blue dye in the proper proportions it is possible to produce all dull colors on dyed fabrics. It is thus possible to express dull colors in terms of amounts of the three primaries needed to duplicate them. In colorimetry this fundamental method is used. A color is expressed in terms of three primaries which have been standardized by physicists and psychologists internationally.[3] It is noticed in practice that secondary colors produced by mixtures of primary dyes are frequently duller than secondary shades produced with individual dyestuffs. When physicists ran into this difficulty, they invented imaginary primaries which would produce all shades in theory. These primaries can no longer be called by color names since they are imaginary. They are called merely X, Y, and Z. One method of expressing colorimetric data is to state the amounts of these imaginary primaries, X, Y, Z, that are needed to match the sample. This method is not widely used. Instead, further calculations are made from X, Y, and Z.

X, Y, and Z can be calculated from the VS curve.[2] There is some diversity of opinion as to the best method of calculation[4] and Ingle[5] has pointed out that accurate calculation is not as simple as once thought. A 19 selected-weighted ordinate method involving summing the ordinates listed in Table IV is recommended for textiles in the event that mechanical integration is not available.

Having X, Y, and Z, it is next customary to convert them to three other values x, y, and Y. This is a mere matter of convenience to the physicist as it enables him to plot all colors of the same Y values on a chart of x and y values as shown in Figure 1. Some color specifications have been set up on this basis.[6, 7]

When this is done it is found that two blues which can just be differentiated visually are separated by a small interval while two oranges which are also just visually differentiated are separated by a large interval. In an effort to make equally different colors plot equally apart as points, methods of distorting the chart have been developed.[8] The distorted plot most widely used is probably that of Judd,[9] who converts the three values x, y, and Y into three more values α, β, Y and plots them. Figure 2 shows how pairs of equally distinguishable colors plot equally separated on this new diagram.

Using these values Judd obtains a difference formula.[10] The magnitude of a color difference can be determined by substitution in Judd's formula.

Going back to the plot of x, y two more color terms are derived. These are dominant wavelength (λ) and excitation purity (p). If a gray sample is plotted, it falls near the center of the diagram as in Figure 3. If the rainbow colors are plotted they fall in a horseshoe curve around the edges. Imaginary spokes are drawn from the central gray to the horseshoe. If a color plots on a spoke to the 500 mu rainbow color, it is said to have a dominant wavelength of 500 mu. Its purity is the percent of its distance along the spoke away from the gray. Thus a purity of 75 per cent would indicate a sample three-fourths the distance out the spoke. Each x, y plot applies only to samples of a given Y value, known as brightness. As a result of this, we now have three new color terms, brightness, dominant wavelength, and purity. These are the colorimetric terms derived from VS data most commonly used.[11]

The dyer has no universal language to describe color. This is due partly to the fact that usually he is interested in color differences not in a description of one color. Speaking of two blue samples and describing a difference he may say one sample is "heavier," "redder," and "duller" than standard. Although the correspondence is not exact, "heavier" is comparable to colorimetric brightness, "redder" is comparable to dominant wavelength, and "duller" is comparable to purity.[12]

As a summary of the colorimetric process, in order to describe a color to a dyer, a physicist gets a VS reflectance curve of the sample, calculates X, Y, Z calculates x, y, Y, interpolates from a chart λ, p, Y, and interprets them in the dyer's terms. In order to express a color difference a physicist gets two reflectance curves, calculates X, Y, Z, calculates x, y, Y, calculates α β Y, and solves for Δ E in the difference equation.

Since a color may be better understood if it is seen rather than described, it is convenient to have on hand an album of colors of known colorimetric values. Thus if a color is described as having certain values of λ, p, Y, it is handy to be able to open up a shade card and find an actual sample of corresponding color. Such data have been calculated for the Munsell Book of Color[13] and are in process for the Shade Card of the Textile Color Card Association of the United States.

The question may be asked, will a knowledge of these color systems and nomenclatures[14] be of assistance to a dyer. In the writer's opinion, from the viewpoint of the dyer's daily problems of color matching, these terms are not particularly helpful. Even if a dyer knew exactly how his sample differed from the standard he was matching in terms of these three indices, this would be of no practical help because it is impossible to add a dyestuff which will change one of these three indices without changing the other two. Thus, in dyeing a piece of wool yellow, increasing the amount of yellow dye used will increase the dominant wavelength, decrease the brightness and increase the purity all simultaneously and in a complex manner.

The terms may find some use in helping dyers arrive at a universal color language. Also the system will probably find increased usage because it is ideally suited to establish permanent color standards and establish color tolerances. Thus, the official red, white and blue colors of the American flag are specified in these terms,[7] and regardless of the way any physical standards may change, the shade of red, white and blue originally accepted is accurately known and may be scientifically reproduced. Thus, at present these terms are useful in discussions of standards

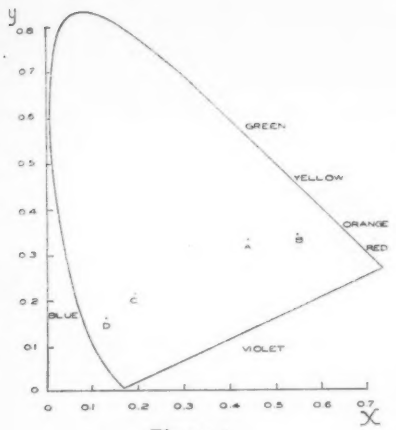


Figure 1
Chromaticity Diagram

A, B, C, D represent four samples. The visual difference between A and B is equal to the visual difference between C and D but the linear separation of A and B is greater.

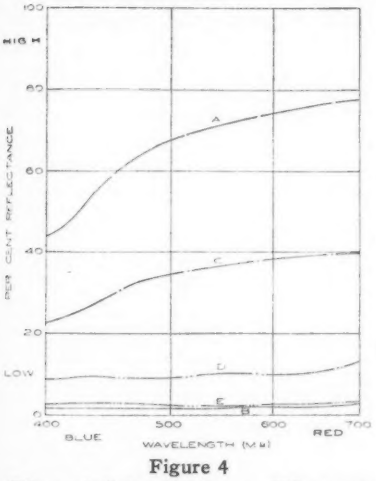


Figure 4

Effect of Color Mixing on VS curves
A. White Wool
B. Black Wool
C. Reflectance of A & B in equal proportions on spinning disc.
D. Reflectance of A & B in equal proportions as blended fibers.
E. Reflectance when dyed up at half strength of B.

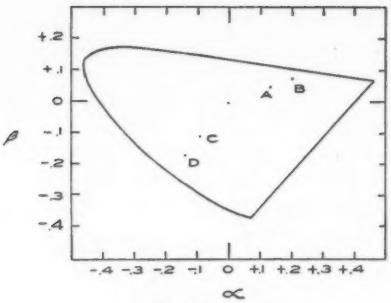


Figure 2
Judd's Diagram

The visual difference between A and B is equal to the visual difference between C and D. On this plot the points are equally separated in each pair.

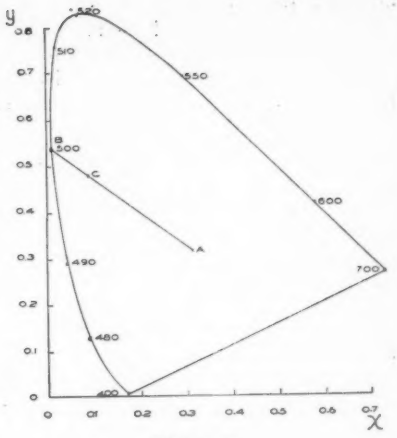
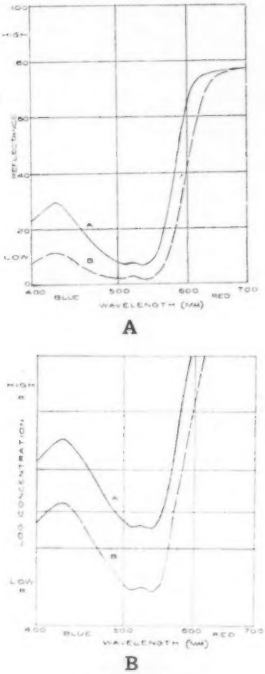


Figure 3
Chromaticity Diagram

Sample C has a dominant wavelength (λ) of 500 mu because it falls on the line drawn from the neutral gray (point A) to the spectral blue of 500 mu (point B). Sample C has a purity (p) of 75% because it is 75% distant from A toward B. All points plotted on the diagram have the same brightness (Y).



A

B

Figure 5

Comparison of reflection and log strength plots.

A and B are dyeings of the same dye at different concentrations.

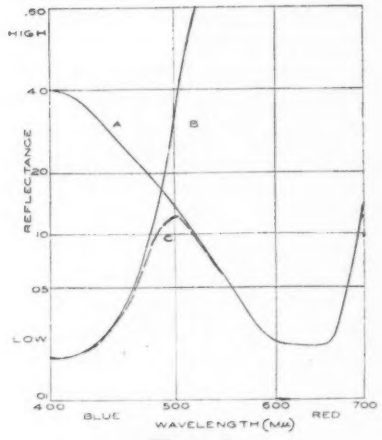


Figure 6
Color Mixing

A. Blue Component
B. Yellow Component
C. Resultant Green, predictable from A and B

and tolerances but they are of no assistance in color matching.

QUANTITATIVE ANALYSIS

Having a VS curve it is also possible to state to some extent what dyes and how much of each was used. This is analysis. Each dye has a characteristic curve shape which is its signature. Each dye added to a sample, changes the curve shape in a predictable way.

If a dyer puts a heading of black wool yarn and a white heading in a whizzer type of hydro-extractor, and the whizzer is spun, the resultant color is gray. Figure 4 curves A and B represent white and black wool. Curve C is the curve of the gray resulting from observing them in a spinning whizzer. If the wool is torn apart and the fibers blended together in a slubbing, the gray resulting from mixing them will have curve D. If the dye in the black is removed and then distributed equally in all the fibers, the gray will have a curve E. Hence in calculating the effect on a curve of the addition of dye it is necessary to consider the manner of addition as well as the amount.

The effect of addition of dyes must be calculated on the basis of an "additive function." In the case of the spinning whizzer the function is R where R is the reflectance of the sample at any one wavelength. In the case of the wool

fiber mix the function is

$$\frac{1 - R}{m(R - 1) + 1.} \qquad 15$$

In the case of dyed wool the function is

$$\frac{(1 - R)^2}{R} \qquad 16$$

Appropriate consideration must be given to surface reflectance.

This means that if one has two dyed wool pieces as references, and wishes to predict the VS reflectance at one wavelength of a piece of wool having both dyes present, it is necessary to substitute R of each reference sample in the additive function, add these two numbers and calculate the new R. In practice the analyses are simplified if the ordinates of the VS plots are the logarithm of the additive function and are done graphically according to the methods first proposed by Weigert[17] and elaborated by Pineo.[16, 18]

Dyes may be recognized by their curve shapes if they are plotted on a log concentration cam.[19] Figure 5 illustrates the usefulness of the log concentration plots in identification work. Curve A and Curve B are the curves of the same dye prepared at two different concentrations. Figure 5a is on a linear reflectance basis and it is not immediately obvious that the two dyes are the same. Figure 5b shows the same samples measured on the log strength basis and it is immediately obvious that the two curves are identical but that one is displaced vertically by an equal amount at each point.

Figure 6 shows how a yellow and a blue combine. Curve A represents the curve of the blue component; Curve B represents the curve of the yellow component, and the result of mixing these two dyes can be predicted graphically. The VS curves thus make it very evident that when a blue dye and a yellow dye are added one normally gets a green. Except in rare cases where the dyes react with each other, the predicted curve agrees exactly with the measured curve.[41]

Just as one can predict the curve of a mix from the curve of two components it is also easy to reverse this process. If a mix is given, it can be analyzed for the components that are present. For instance, given a brown represented by curve "A," "B," and "C" in Figure 7, examining this curve one immediately recognizes the characteristic signature of Alizarin Saphirol B (C.I. 1054) as given at the absorption maximum near the letter "A." Drawing in a curve of Alizarin Saphirol B of known concentration, represented by the curve "FA" and subtracting it out from the mixture, gives "C," "B," "D." Looking at this resultant curve "CBD," the signature of Amido Naphthol Red G (C.I. 31) in the characteristic absorption at point "B" is recognizable. Subtracting out curve GBD, which is the curve of Amido Naphthol Red G only curve CE is left. Curve "CE" is found to agree with the curve of Xylene Light Yellow (C.I. 639). In this way by subtracting one component after the other graphically, it has been possible to determine by analysis which three dyes were present and in what proportions they were present to give the brown.

Thus far we have considered the interpretation of VS data and have briefly outlined the colorimetric and analytical procedures. Now let us consider the applications in a textile mill.

COLOR MATCHING

One of the most obvious opportunities for application of a VS in a textile mill is in color matching. The best answer to whether or not the VS can be used in color matching is Table I. Nine samples were submitted to be matched and were measured on the VS. These nine curves, together with three curves of dyes to be used in matching, were submitted to a laboratorian. The laboratorian, without seeing the standards to be matched, and using different dyes than were used in the standards, calculated matches. The accuracy of the matching may be seen by reference to Table I, which lists the visual difference in Judd units. Differences under 2 are fairly small visually. This was accomplished by a combination of analytical and colorimetric methods.[20] The time required for calculation of a match varies widely depending upon the closeness of match needed, the similarity of dye curve shapes in match and standard, and the skill of the operator of a calculating machine. An average time might be 30 minutes.

TABLE I
Evaluation of Color Matches

	Sums of 100 Illuminant A Ordinates						*Visual*
	Standard			*Match*			*Difference*
	X	*Y*	*Z*	*X*	*Y*	*Z*	*Judd Units*
A	1408	1249	815	1493	1316	880	1.3
B	451	467	700	462	482	712	1.7
C	2106	1901	1510	2210	1997	1567	1.3
D	2081	1905	815	2283	2126	938	3.6
E	1175	1155	1244	1138	1124	1276	1.5
F	1492	1485	1861	1396	1366	1834	2.1
G	398	492	836	468	565	914	2.1
H	2224	1426	517	2406	1577	629	2.7
I	6463	6106	767	6963	6567	765	3.6

One reason why matches are not perfect may be failure of the theory of prediction. In general it has been found that except in a few cases of dye interaction the theory holds very well.

A second reason for the differences may be dyeing randomness. This randomness, which may be due to variations in the quality or origin of the stock[15] or variations in dyeing conditions[21] results in a different shade than would be predicted by even the most exact theory. In order to test out formulating with a VS the following experiment was devised. Twelve samples of wool were dyed up in one laboratory. These samples contained three dyes in various proportions. These samples were submitted as unknowns to another dye laboratory to be matched and were also submitted to the VS for analysis. When the work was completed, three sets of figures were available: one, the true dye content; two, the VS analysis, and three, matching formulations arrived at by conventional dyetest procedures. Inspection of the figures showed that the VS analysis was

closer to the true dye content than the dyetest analysis. But inspection of the dyeings of all the formulations showed that the dyetest matches were closer visually. Because of dyeing randomness the dyer had produced a closer match with a new formulation than he would have had by repeating the old. This confirmed what had previously been reported[22] that the limiting accuracy in prediction of color is the dyeing process rather than the measuring method.

A third reason for the observed differences is that different dyes were used in the matches than were used in the standards and even a perfect match made in this way is only perfect to one observer under one illuminant. This is illustrated by the following test. A taupe was made up of a red, yellow, and blue. It was decided to attempt to match this in tungsten light with an orange, a violet, and a green. The three components were measured and the final formulation calculated. When dyed every one agreed it was not a match. Some thought it was too red, some too blue, some too green. Choosing two dyetesters who deviated widely on the Glenn Colorule[23] and asking them to prepare a match gave the following figures:

TABLE II
Parts by Weight of Dye Required

	Colorist A	*VS*	*Colorist B*
Orange	0.226	0.197	0.190
Violet	0.152	0.164	0.160
Green	0.070	0.070	0.070

Each colorist thought that the other two matches were unsatisfactory. The theoretical colorimetric observer merely said the VS match differed from standard by 1.8 Judd units. This is as close as can be expected unless unusual care is taken in dyeing and measurement.

Does this mean that since reasonably good formulations can be made with the VS there is no need for a mill to have a dyer? No, because in the cases described, the matching problem was quite simple. In other cases a dyer can handle a matching problem better than a VS. For instance in matching a wool cloth to a plastic button a dyer is necessary. In matching a fluorescent shade the VS confuses the fluorescence with reflectance. In union goods the dyer can examine the individual fibers and make additions to correct the shade of one or the other type of fiber. The effect of weave makes it impossible to give an exact VS match unless the standard curve is on the same fabric. The VS is unable to choose dyes of the proper fastness properties. The VS can be used as a tool to assist the dyer but cannot be used to replace him.

EVALUATION OF COMPETING DYES

Another application in the mill is the evaluating of competitive offerings. One difficulty is that if the randomness in dyeings and VS measurement is such that one datum is accurate to $\pm 5\%$ in concentration, no difference less than 7% may be considered real. Thus the average of a number of dyeings must be taken if greater accuracy is desired. A second difficulty is that the relative strength of dyeings may change or even reverse at different dyeing conditions. Thus the data are only useful if the laboratory test duplicates the mill conditions. A third difficulty is that in the event of a shade difference, which is very common among competing types, exact concentration differences cannot be determined. They cannot be determined from the VS curve because different values of relative concentration are obtained at each wavelength. They cannot be determined visually because various observers will penalize concentrations for shade differences in different ways. The fourth difficulty in getting comparisons of samples is that the comparison holds strictly only for a particular formulation and in general a mill is interested in a dye strength for many formulations.

It has been proposed to evaluate competitive samples from solution data.[24] Here in addition to shade difficulties, differences in exhaust and selectivity are important. There are three major corrections that need to be applied to solution data in the study of competitors' types in order to correlate with dyed cloth results. These are:

1. Exhaust differences
2. Selectivity differences
3. Shade strength correlation

As an example of exhaust differences reference may be made to the comparison of two samples of Direct Deep Black EWEx (C.I. 581). Curve A and B of Figure 8 show the solution curves of these two dyestuffs and it is apparent that the B sample is 103% versus the A dye in solution. However, when dyed on cotton by a standard method, 90% of the A dye put in the dyebath goes on the cotton and 10% remains in the bath. In the case of the B dye, 87% goes on the fiber and 13% remains in the bath. This means a correction factor must be used to correct the B dye solution strength when it is compared against the A product. The effect of this correction is to increase the strength of the A solution by 3% and thus as regards solutions corrected for exhaust the comparison is 100 to 100.

The phenomenon of fiber selectivity has been investigated by Millson and Royer.[25] They have demonstrated that the prediction of fiber strength can be thrown off if there is selectivity difference. For instance they dyed a skein with Alizarin Cyanine Green (C.I. 1078) at 150°F. This resulted in many fibers in the cross-section being dyed heavily while others remained undyed. Part of this sample was then taken out and put in a separate bath and boiled with no additional dye put in. This boiling had the effect of leveling out the dye among all the fibers and resulted in the dyeing appearing much stronger to the eye. Here, then, is a case where the initial dyebath was the same and the exhausts were the same, but the apparent strength of the fiber was different. Selectivity differences may occur in products of different manufacture because of the slight differences in chemical structure, or impurities.

The shade-strength correction difficulties are the same as for a comparison of fiber samples discussed above

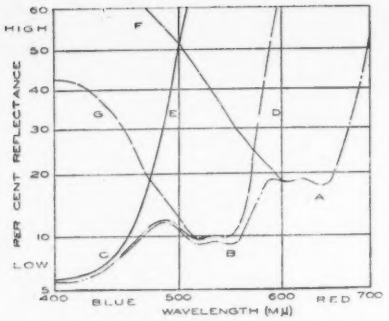


Figure 7

Color Mixing
The components of Brown A, B, C, may be determined.

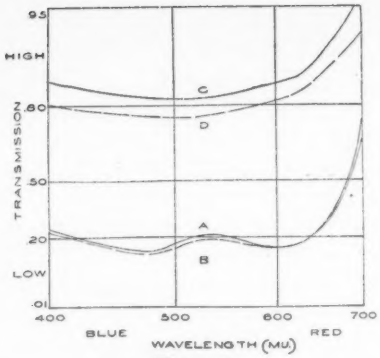
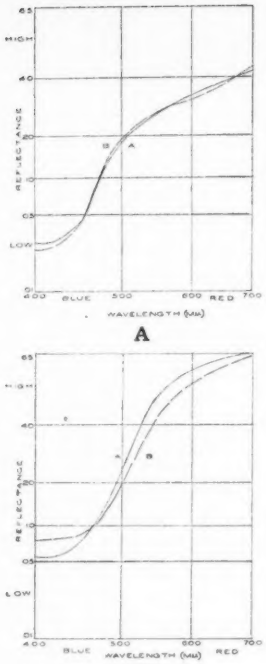


Figure 8

Exhaust Correction
Direct Deep Black EWEx on Cotton
A, B, initial solutions
C, D, exhaust solutions
Part of data needed to predict fiber color strength from solution data.



A

B

Figure 9

Light Fastness Evaluation by Different Observers Four hours in Fade-Ometer.
solid line —— unfaded
dotted line - - - faded

except that they are even more baffling in the case of chrome dyes, developed dyes, and others where the solution color is quite different from the finished color on the fiber.

DYE STANDARDIZATION

Another application is the testing of incoming shipments of dye from the same manufacturer.[21] The uniformity of these shipments is of importance to the dyer because in some combinations a 1% difference in dye concentration is readily apparent visually. This is true for instance in the case of varying the Red concentration by 0.001% in a taupe dyed with 0.100% Amido Naphthol Red C (C.I. 31), 0.06% Alizarin Saphirol B (C.I. 1054) and 0.16% Xylene Light Yellow (C.I. 639). Strength comparisons are often satisfactorily made with solution data because the three correction factors which occur with products of different manufacture are often negligible in this case. Even here there is some uncertainty, for if the supplier should improve his process to reduce the amount of non-substantive colored impurity, the solution data might indicate a weakness which was not borne out by fiber tests.

FASTNESS EVALUATION

Another application is in the evaluation of fastness properties. This may be discussed for light fastness since the approach to the problem is the same for all fastness tests.

Fastness properties are easily and reproducibly measured. A sample of a Sulfur Yellow on cotton was tested in a Fade-Ometer by a dyetester and was rated as 2 in light fastness. The VS curves of Figure 9a show how the curve changed from a solid line of the unfaded sample to the dashed line of the faded sample. Very little change had taken place in the sample. At an entirely different time a different dyetester was testing some acid yellows on wool. This particular dye was rated as having a fastness of 2 also. It is evident from the curves of Figure 9b that a considerable change had taken place in this sample before it was rated as having faded with a fastness of 2. Such widely different evaluations of fastness carried out by different dyetesters at different times can be eliminated by evaluating fading with VS measurements.

Ordinarily color matchers wish to have fading results expressed in terms of hours exposure to produce a just objectionable color change. This expressed desire to evaluate a color change indicates that colorimetric interpretation of VS data is needed.

Several methods of evaluating fadings have been proposed[26] but none have yet found universal acceptance and wide usage. Figure 10 shows a typical fading curve of a dye. It will be noted that at some points the curve rises and at other points it is lowered. This corresponds to a loss in depth and a dulling. In some cases dulling is the predominating change, while in other cases the loss of depth is the predominating change. To be satisfactory, any method of interpretation must treat all such cases. The strictly colorimetric methods of Judd and Nickerson seem most promising although the added accuracy over visual estimation is counterbalanced by the measurements and calculations needed.

On the other hand, much can be said in favor of analytical data. The main usefulness of a tabulation of fastness is in the prediction of compatible combinations, yet colorimetric data are often inadequate in this connection.

(Continued on Page 16)

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"ARMY AND NAVY NIGHT" AT MEETING OF NEW YORK SECTION, DECEMBER 3, 1943.

Report of meeting appeared in last issue. Papers presented will be published shortly.

Finishing with Alkali Soluble CELLULOSE ETHERS and Some Recent Developments in Their Use*

J. ANDREW CLARK
Sylvania Industrial Corp.

CEGLIN is the registered trade mark of the patented alkali soluble cellulose ether manufactured by the Sylvania Industrial Corporation. This product is being used extensively in the production of laundry fast durable finishes. Before discussing this further, it might be well to point out that in general there are two types of durable finishes, in the first of which the physical structure of the cellulose fiber in the yarns is changed by action of various chemicals. While the patent literature contains numerous references to proposed treatments of this type, only two or three have gained any recognized position in the textile field. Such finishes for instance, are mercerization, the Heberlein and some of the cuprammonium treatments.

The second type of durable finish embraces additions to the fabric which by reason of their firm adherence to the fibers are able to confer specific and unique properties with more or less laundry resistant characteristics. Ceglin is outstanding in this later class. While not exactly new this product is one of the more recent cellulosic finishes to receive wide acceptance in textile finishing. The older viscose and cellulosic solutions in cupramonium have been commercialized for twenty years and over with varying degrees of market acceptance. The application of these latter two, however, have been attended with the necessity for large initial investments, delicate technical control and industrial health hazards. These handicaps are avoided in the use of Ceglin. Application of viscose has been especially troublesome, in that, first, the solutions are relatively unstable, which complicates shipping and storage, and secondly, noxious and poisonous gases are evolved during coagulation which constitutes a definite health hazard; these gases also severely corrode any metal equipment within a wide radius.

Use of cuprammonium solutions is only slightly less troublesome, while also being somewhat expensive, and said to be very difficult to control for uniformity of finish.

Lillienfeld, a Polish chemist, first prepared and patented alkali-soluble-cellulose-ether that looked not unlike the original cellulose from which it was made. When suitably dissolved in caustic soda, solutions are obtained by which almost all standard cotton and rayon finishes may be produced with the novel advantage that these finishes are fast to repeated laundering.

The Sylvania Industrial Corporation has acquired exclusive rights under the numerous Lillienfeld patents to manufacture, use and sell these cellulose ethers for textile treating. To suit various requirements "Ceglin" is prepared in four viscosities—6, 2, medium-d· soft-d. The first two of these are used for the production of crisp and firm finishes, the latter two for softer and fuller finishes. Two other types are available for specialized applications.

Dry Ceglin is a white fluffy fibrous material resembling linters in appearance and is dissolved in cold dilute caustic soda to a colorless viscous solution.

These solutions are applied for the most part in concentrations ranging from 1½% to 6% although for some special applications higher concentrations are suitable. The caustic content may also be varied as conditions demand.

Fundamentally, the application of Ceglin requires two steps. The first of these is saturation of the fabric with Ceglin solution of appropriate concentrations and type by pad, mangle, back filler or similiar equipment. After saturation of the fabric, coagulation is required to convert it to its durable or laundry resistant condition. Technically, coagulation, is secured by treatment with acid or by removal of the greater part of the water either by cans or a tenter frame. Other methods of coagulation are available for special finishes and will be described later.

Range installations are peculiarly adapted to Ceglin application, because of the two step character of the operation. Thus for many fabrics a mangle in range with a three or four box machine for acid coagulation and washing at width is ideal. In other installations where stiff crisp finishes are desired, a set of dry cans following a mangle has worked out very well, or if desired a tenter may follow the mangle. Obviously range operation is not imperative and if desired the cloth may be batched on a roll after saturation with the Ceglin and then finished on a jig. Certain fabrics may be treated with Ceglin in a mangle and then coagulated in rope form if care is taken to prevent excessive pressure forming rope marks during coagulation.

Mineral fillers and pigment colors may be applied with Ceglin either by a mangle backfiller or coating blade. Ceglin forms an excellent binder for these, and for the first time a laundry-fast backfilling may be obtained. Ceglin may also be used as a thickener for printing.

Considering the foregoing remarks in the nature of an introduction it may be of interest to describe briefly the preparation of the alkali soluble cellulose ethers and

* Presented at meeting, Southeastern Section, November 22, 1943.

method of their solution for textile application. Without boring you with the details of the patent literature or the prior art; Lillienfeld first taught the preparation of the alkali soluble cellulose ethers and pointed out the possibility of their use in textile finishing. The preparation is not unlike that for viscose solution in that cellulosic material such as spruce pulp or purified cotton linters is converted to alkali cellulose by use of strong caustic soda. After appropriate aging, this alkali cellulose is treated with an alkylating agent. Various alkyl compounds have been employed to produce the different types of cellulosic ethers possible, although for the manufacture of the hydroxy ethyl cellulose ethers, chlorhydrin or ethylene oxide is used. Reaction of these latter produce the ether, without much visual change in the physical appearance of the original cellulosic material. The reaction mass is then freed from the caustic soda or soda and salt by hot water extraction after which it is dried.

The preparation of the cellulose ether is modified and controlled in various ways to obtain the several types and viscosities required for industrial applications.

Variation in the ratio of the combined hydroxy-ethyl group to the glucose nucleus in general effects the solubility of the finished product in caustic soda. On the other hand, the Viscosity is modified by the degree to which the cellulose has been degraded. This degradation may be produced during the alkali-cellulose aging, or following preparation of the ether. While no appreciable difference is observed in the final product the latter method is preferable because of manufacturing consideration. In general, this phenomenon of degradation accompanied by lowered viscosity is familiar to most plant chemists who use this relationship in measuring effects of chemical action on cotton piece goods and yarns.

Solution of "Ceglin" takes place in caustic soda at reduced temperatures, generally in the range of 40 to 32° F, but following solution the liquid may be warmed to room temperatures for application. If desired the solution may be applied as high as 140° F, although at temperatures above 120° F, surface evaporation from the pad box may introduce difficulties from skin formation, if all parts of the surface is not agitated.

Two methods are available for dissolving; either direct cooling in a brine jacketed cooler or by addition of finely cracked ice to the mix.[1] This reduces the temperature sufficiently for solution to take place.

Concentrations of Ceglin may be prepared containing up to 10% to 12% with caustic soda of like concentrations. Beyond these values gel formation commences. If the caustic concentration is within the range of that used for mercerizing, during application the Ceglin may be coagulated. One type of Ceglin shows this effect especially well and may be applied simultaneously with mercerization for treating some fabrics.

Solution by the ice method is simple and can be easily carried out in a cylindrical steel shell fitted with a gate type agitator running at 20 to 30 R.P.M. Almost any type of improvising equipment has been used, and for the most part have been assembled from idle equipment by plant mechanics.

Three steps are required for solution.

1. Wetting out of the Ceglin with water to expel air from any dry hard lumps and to facilitate mixing.
2. Addition of the required amount of caustic soda solution with thorough stirring.
3. Addition of the finely cracked ice. Generally 30% on the weight of the total mix is required to produce a satisfactory drop in temperature.

The description of the procedure for a typical mix of 3000 lbs. 6% Ceglin in 8% Caustic soda may be of interest.

1200 lbs. or 144 gallons of water is run into the mixer to which is added 180 lbs. of shredded Dry Ceglin or in small lumps while stirring. This produces in five to ten minutes a uniformly wet mush.

720 lbs. of 63¼ gallons of 33% caustic soda is then run in as a thin stream while continuing the stirring. This swells the fibers greatly, and produces a translucent effect resembling solution. Examination in the microscope however, would show this otherwise. Some heat of dilution is produced by the caustic soda, but this is compensated for by the ice. If equipment is available the mix could be allowed to cool overnight before proceeding if desired. Stirring is continued until a uniform mixture with the caustic is obtained.

900 lbs. of finely cracked ice is then added, as nearly at one time as possible while stirring is continued. The bulk of the ice dissolves in the first five minutes, and at the end of a half hour, maximum drop in temperature has been reached with practically all the ice having dissolved.

The solution as first prepared has an abnormally high viscosity due to the lower temperature.

While no stoichiometric relation is known to exist between the concentration of Ceglin and caustic soda in a given solution, we do know that when the caustic content equals or exceeds that of the Ceglin, the solution may be diluted with water without gel formation. When the caustic content on the other hand is less than that of the Ceglin, gel formation proceeds slowly, and at the end of a few hours the mass may be solid.

Ceglin solutions may be applied to a variety of fabrics and constructions in all manner of finishes, and is perhaps with the exception of starch the most versatile of finishes as yet offered. The finishing of cottons, spun and filament rayons is naturally the most logical, but other fabrics such as acetates, jute ad wool can also be treated if proper consideration is given to the action of alkaline solutions on these materials.

The first commercial activity was naturally directed towards the finishing of volume white goods. Of these,

[1] Process Patented.

wide and narrow bed sheetings and table damask was finished quite successfully. For economic reasons the 64 x 64 quality seemed best suited for a durable finish and on this quality an excellent result was obtained. The Ceglin in one plant is applied to the kier boiled and dried cloth after which it is bleached and finished in the usual manner. This routing is a little unusual, but was best adopted to the equipment lay-out of this plant and necessitated less changes than any other routing would have. Generally for whites it is more economical in operations and cost to apply the Ceglin to the grey cloth, then followed by acid coagulation, and kier-boiling.

Application of Ceglin to grey cloth, however, necessitates that the grey is well penetrated by the Ceglin solution. The usual set-up of a pad or mangle fitted with box having one emersion roller is occasionally insufficient. A larger box with two or three dips is then far better and usually more satisfactory.

Table damask has been finished in several ways, using both the 2 and 6 viscosity products in 4 to 6% concentration. Whereas one plant elected to run this on the bleached and mercerized cloth, others have treated their goods after kier boiling and mercerizing. There is an additional possibility that these fabrics may be saturated with Ceglin then coagulated and mercerized simultaneously with strong caustic with a slightly reduced operational cost.

The cloth is later calandered and shrinered. Lustrous linen-like, crisp finishes are produced which retain much of their crispness and lint free characteristics after many launderings. While these factors are of value in detail domestic sales, they are much more important to laundry supply houses and institutions who operate their own launderies, by reason of the accumulated saving in starch throughout the serviceable life of the article.

The finishing of interlinings for obtaining a finish that will retain a high degree of stiffness after repeated launderings is of interest. U. S. Army Specification No. 6-291 requires the cloth to have a clear smooth finish which will not show a loss in stiffness of more than 60% after 5 launderings in accordance with the procedure of CCC-T-191a. Ceglinized interlinings, however, have been finished to meet satisfactorily the earlier required minimum of not more than 35% loss.

Satisfactory results have been produced by semi-dry coagulation of Ceglin applied to either grey or bleached cloth. The finish differs slightly, in that when equal Ceglin concentrations are applied to the grey cloth, the resultant finish is somewhat softer initially than when the Ceglin is applied to the bleached cloth. The breakdown after the first laundering, however is slightly more for the latter finish. Semi-drying produces a sheer, lustrous, linen-like crisp and somewhat open fabric. In addition, the fabric usually shows reduced shrinkage after laundering.

A pad or mangle in range with 40 odd cans works out well. If desired, a frame may be used in place of the cans, or a combination of cans and frame. The cloth is padded with 4 to 6% Ceglin and then semi-dried. We suggest that approximately 75% of the water be removed at this point. The cloth then acquires a firm wiry character with loss of any characteristic slippery or caustic feel. It is then soured to remove the alkali after which it is handled as usual, except that no starch is used. Tinting may be incorporated with the Ceglin by addition of suitable blue pigment colors. The non-retention of chlorine in laundering and ability to retain its white color are very desirable properties of Ceglin interlinings which gives these fabrics a superiority over those finished with many of the resins or casein-formaldehyde.

An interesting variation in finishing with Ceglin to produce a crisp wiry finish for use as a metal screen replacement is being used in Canada. In addition, the cloth must retain a considerable degree of stiffness while wet. This effect is produced by first treating with casein-formaldehyde and then with Ceglin. The resultant finish is more resistant than if either were used alone.

The above illustrations are typical of white goods treatment. Mineral fillers extend the range of application to building up print cloths, dress goods and other constructions, and are not only made laundry resistant and durable but may also be used for coloring.

Extremely large yardages of O D insect cloth, head nets, camouflage cloth, etc. has been and is being simultaneously finished with Ceglin and dyed with pigment colors. While any pigment which will withstand the action of the caustic soda and acid used in the process may be used, best results require a proper selection of pigments for type and grinding. In this way, ease of penetration, uniformity and increased depth of shade at best money value may be obtained. The conditions surrounding the use of pigments are quite different from those connected with dyeing. Initially, some considerable amount of research was involved to master the art. While we have stressed O D, the colors are by no means limited to these shades. The range in depth may extend from one part of pigment paste to 3000 of Ceglin solution to the heavier shades using one part of pigment to four of Ceglin solution.

The fastness of these combinations to laundering, light chlorine and cocking is for the most part good. Like dyestuffs however, the pigment colors also show individual characteristics which permit selection within a fairly wide range to secure a type most suitable for the service intended.

There are, however, some limitations to the use of pigments in Ceglin. Whereas light and medium shades are reasonably fast to laundering and crocking, the heavy shades ordinarily are not. Uniformity of shade with no tailing off is also a decided advantage.

It is imperative that the cloth intended for pigment dyeing with Ceglin be uniformly absorbent and that in padding no creases or wrinkles pass through the nip. Pigments unlike direct colors have no tendency to level on the roll after batching, consequently, the pigment must be applied most uniformly. Range operation is most satisfactory but

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if necessary the goods may be batched at width and then acid coagulated open. Care must be taken, however, that no drag occurs. Under no circumstances should the fabric be put into rope form for coagulation or washing where squeeze rolls are involved. Light weight fabrics such as laces may be run in reel machines after souring. Wetting agents should be used with caution. These products vary so greatly in their surface activity, that their effect on the pigment dispersion should be studied before use. Excessive amounts or unsuitable types may cause unexpected results, either in change of shade or production of a color effect best described as an eggshell appearance. The cost of application is moreover attractive, especially where range operation can be employed.

We now pass to one of the more interesting and revolutionary developments in the application of Ceglin, namely warp sizing.[2] As you all know, warp yarns for the most part and especially single ply warp yarns require reinforcement and protection to withstand the tension and chafing to which they are subjected in weaving. This has been accomplished for a long time by the use of faraginous mixtures, in the slashing. With the exception of a few fabrics, such as pocketing and certain linings, this size must be removed during finishing.

The necessity for steeping is thus eliminated when the warp is sized with Ceglin.

Some changes, however, are required in the conventional warp sizing slasher in order to apply Ceglin. The present installations consist of a shallow four box machine installed in place of the usual starch box, between the creel and the drying cylinders.

The boxes of this machine are:

First: A Ceglin saturation box and pad,
Second: Acid coagulation box,
Third and Fourth: Wash boxes.

These boxes have all been designed shallow to facilitate the operator working around the yarns as well as to prevent the tendency of the yarns to group together. These are fitted with appropriate submersion and squeeze rolls.

The design of the unit has been guided by the following basic considerations:

1. The grey yarn must be uniformly saturated with Ceglin.
2. The impregnated yarns must be coagulated in acid without being in contact with each other.
3. Thorough washing must be provided.
4. Bunching or rolling of the yarn must be avoided.

By so splitting the warp as it enters the Ceglin solution that alternate ends pass under different submerged rolls, uniformity of saturation is considerably improved. Likewise it is necessary to provide pad or mangle rolls which provide for uniform and reproducible application. Unlike starch sizes, the Ceglin size carries through to the final fabric finish, consequently it is quite evident that the uniformity of application must be of the same order as that of the finish expected. We therefore suggest use of a pad or mangle having a bottom metal roll, with a rubber covered top roll of medium density, with pressures of 75 to 100 # per linear inch.

In passing to the acid for coagulation, it was first observed that the surface fibers or fuzz on the yarn stuck and partially cemented together, and if at the same time the yarns bunched, excessive breakage resulted in the leashing after drying. The solution was to separate the yarns prior to entering the acid. This not only eliminated the cementing but produced a cleaner and smoother yarn.

The yarn is then thoroughly washed by passing through the two boxes fitted with squeeze rolls. Warm water sprays added to the nips are quite helpful. To prevent yarn rolling in the various boxes it is suggested that a grooved metal roll be mounted in front of each pair of squeeze rolls. It is thought that if the grooves are 20 or more to the inch, serious rolling will be avoided.[3]

After washing the yarn is then dried on the conventional type of cylinders, leashed and beamed.

True, Ceglin slashing is more complex than slashing with starch, but it offers much more opportunity for improvements in the fabric, as well as weave room practice. In the first place, increased warp tensile strength in the order of 10 to 35% has been reported.

In appearance the yarns are smoother, more dense and lustrous. Singeing thus does not need to be as close. They are, however, somewhat more flexible than starch sized yarns, and consequently the usual standards for stiffness used by some slasher men to determine weavability do not apply.

Insufficient reports have been received to make much of a claim for increased weaving efficiency. We do have indications, however, that for the most part it is about the same and in some cases slightly better than starch slashing.

Lower weave room humidities of 65%, however, are helpful in not only promoting increased weaving efficiency but also in retarding rusting of reeds and other exposed metallic parts. Improved working conditions, moreover, result indirectly through the lowered humidity.

Fabrics woven with Ceglin sized yarns show increased abrasion resistance. The amount is dependent on several factors such as weave construction and concentration applied. Increases of 100% have been noted in some twill constructions.

Shedding in the loom has not been thoroughly investigated as yet but indications are that when the loose warp fibers are laid properly, very little shedding occurs. On the other hand, if the yarns are permitted to adhere during coagulation and then broken apart, the loose fibers then may break off in running through the loom.

Various other advantages have been reported but as yet these can only be considered as indications. For in-

[2] Patent applied for.

[3] Patent pending for apparatus.

stance, the resultant cloth gives a wider loom width when reeded the same as starched warps. This should be a decided advantage in reducing if not entirely eliminating temple chafing. This wider width may also account for reports that such fabrics produce a better width after the Sanforized wash test. We also have reports that after Sanforizing there is a net gain of approximately 1% in finished yardage over that shown by fabrics with starched warps.

Where dyeing is required, the usual methods are applicable. These goods have been dyed in both commercial and vat colors satisfactorily. In fact, vat dyes and twills may even appear more lustrous and show less appearance of a fuzzy character. Where cross dye effects in medium and light depths are desired, pigment colors may be applied simultaneously with the Ceglin. In fact, we visualize the possibility of preparing warps with single pigment color stripes by use of two application boxes, one for clear Ceglin, the other for colored Ceglin.

The finishing of rayons, both filament and spun shows considerable interest. In order to produce a mellow and full hand, we have developed two low viscosity types namely Medium and Soft D. Low percentages in the order of 2 to 3% have remarkable bulking effects, and when finished off with small amounts of cationic softeners, delightfully soft, lofty, wooly effects can be obtained.

Crisp firm finishes likewise can be obtained suitable for finishing slacks, brassiere and corset cloth, by use of regular type Ceglin of Viscosity 2. Increased concentrations of Ceglin, however, has a tendency to produce a dense papery effect. This generally limits the amounts applied to 3 to 4%.

As with cottons, both acid and the semi drying method may be used for coagulation. The later, of course, producing a somewhat crisp effect. Acid, however, should be used for all soft finished.

Clay and other pigments can be used to advantage in finishing pigmented taffetas. These are added to the padding solution and applied in the usual way. One to two times the percentage of Ceglin may be used both as a filling and delustrant. The fastness to laundering is excellent.

For the most part, the most economical application consists of padding in the grey, followed by acid coagulation at width either in a box or in a jig. The cloth is then boiled off in the usual manner, dyed and finished. Should semi drying be involved the grey cloth is padded and passed directly to a low temperature frame. Care should be taken to avoid over heating or over drying to avoid fabric damage.

Spun rayon with acetate effects may be finished for cross dyeing successfully. On first thought, the question of saponification of the acetate arose. This is not a serious handicap in that saponification is a function of time, temperature and caustic concentration, and in this operation, each of these factors is maintained at a minimum. The cloth is padded and coagulated at room temperature, the time is short. and the caustic is rarely over 3%. Numerous laboratory checks have confirmed this but most imposing has been the fact that some very considerable yardage has been run commercially. The laundry fastness however, is not quite as good on all-acetate, but nevertheless is quite imposing. Some all-Celanese table damask padded with 2% Ceglin in the grey, after boil off and dyeing retained the finish for five launderings quite well. A word of caution, however, whereas Ceglin may for some finishes be coagulated by passing through boiling water, due to removal of the caustic soda, this method should not be used with acetate, or saponification will be extensive.

MEETING, PHILADELPHIA SECTION

A MEETING of the Philadelphia Section was held on December 10, 1943, at the Hotel Philadelphian. 88 persons were present.

Eric T. Laurin, Director of Textile Service, Calgon, Inc. was the speaker of the evening. His subject was, "Calgon in the Textile Mill," and his talk was supplemented by motion pictures in color. This proved to be most interesting and instructive. After the lecture, questions were asked by several of the members, which were satisfactorily answered by Mr. Laurin.

The following names were presented by the Nominating Committee, and were duly elected for the year 1944 as officers of the Philadelphia Section.

Chairman Emeritus—Elmer C. Bertolet

Chairman—Boyce C. Bond

Vice-Chairman—Arthur W. Etchells

Treasurer—Curt R. Baeringer

Secretary—A. E. Raimo

Sectional Committee—Walter F. Fancourt, 3rd, Miles A. Dahlen, Richard B. Stehle, Dr. Edwin C. Dreby

Councilors—Arthur E. Jones, John F. McCoy, James P. Conaway.

Respectfully submitted,

ROYCE C. BOND, *Secretary*

MEETING, NORTHERN NEW ENGLAND SECTION

THE meeting of the Northern New England Section originally scheduled for January 21, will be held on January 28th at 6:30 P.M. at the Hotel Manger, Boston. Massachusetts.

The speaker will be Dr. Walter S. Landis, Vice-President and Director of the American Cyaramid Co., who will speak on "The United States of America as the Post War Chemical Center of the World."

UNEMPLOYMENT REGISTER

Members of this association, who are without employment, should forward a statement as to their qualifications and experience. This information will subsequently appear on these pages with an identifying key number. Employers are also requested to file with the American Dyestuff Reporter any vacancies which may occur in their business.

NOTICE TO EMPLOYERS

Personal Histories and Employment Records of the following applicants are on file at the office of the American Dyestuff Reporter. Prospective employers may examine them without obligation.

Key: A—Laboratory and research; B—Dyeing or printing; C—Finishing; D—Sales; E—Instruction; F—Executive.

A-2

Education—Graduate, Lowell Textile Institute, B.T.C., 1933.
Experience—Experienced as textile chemist, chemist-spotter, agricultural chemist and U. S. Army inspector on cotton, rayon, woolens and worsteds. Seeks research or control position with future. New England preferred.
Age 34; married; references.

A-3

Education—B.S. in chemistry, Lehigh University.
Experience—Experimental engineer and analytical chemist, 8 mos.; assistant superintendent of silk corporation, assisted in planning and directing production, in charge of laboratory, technical work, personnel, purchasing, etc., 2 years; inspector of ordnance material, to the present. Seeks position as chemical engineer or chemist.
Age 26; single; references.

A-B-C-F-3

Education—High school and full apprenticeship.
Experience—Practical Dyer and finisher. Long experience on specialties for men's wear trouser trade in cottons, cotton and rayon mixtures and cotton and Celanese. Is disposing of interests in progressive finishing plant and seeks position as manager, assistant manager or technical advisor. Thoroughly understands requirements of Quartermaster, especially on herringbone constructions.

C-1

Experience—Experienced as assistant finisher, finisher and superintendent of finishing since 1923. Experience on automobile cloth, boucle overcoatings, high grade men's wear worsted, low grade woolens, medium grade plaid-back overcoatings, fleece finish overcoatings. Bolivias, kerseys, medium-grade men's piece dye suitings, uniform cloth, flannels, ladies' dress goods and coatings, government serge and elastique fabrics, marine wool, flannel shirtings.
Age 37; married, 3 children; references.

C-2

Education—Completed course in textile engineering and chemistry except for final half-year at Clemson College.
Experience—11 years of experience in finishing operations, including 8 years as overseer. Experienced on all types of cotton cloths. Experienced on pure finishes, backfilling, Sanforizing, pre-shrinking, and application of several types of water repellent finishes. Seeks position as overseer of finishing department.
Age 32; married; references.

CALENDAR OF COMING EVENTS

Meeting, Philadelphia Section, Hotel Philadelphian, Philadelphia, Pa., January 14, 1944.

Meeting, Council, Chemists' Club, New York City, January 21, 1944.

Meeting, Research Committee, Chemists' Club, New York City, January 22, 1944.

Meeting, Rhode Island Section, January 28, 1944.

Meeting, Northern New England Section, Hotel Manger, Boston, Mass., January 28, 1944. Speaker: Dr. Walter S. Landis, American Cyanamid Co.

Meeting, New York Section, Downtown Athletic Club, 19 West St., New York City, February 4, 1944.

NOTE: Date of April meeting has been changed from the 7th to the 21st.

OBITUARY

ROBERT D. HOWERTON

ROBERT D. HOWERTON, for the past two years western sales manager at Chicago for the Calco Chemical Division, American Cyanamid Co., died recently. He had been a member of the sales staff for a number of years.

Robert D. Howerton

Mr. Howerton was born in Lexington, Virginia, and attended public schools of that city. He received his B.S. degree from Washington and Lee in 1922. Prior to his association with Calco, he had been identified with the textile dyeing and finishing business in the South, and for some time after joining Calco, he was a sales representative in that territory.

Mr. Howerton was a senior member of the Mid-West Section of the American Association of Textile Chemists and Colorists.

He is survived by his wife, two sons and a daughter.

WILLIAM H. STANTON

WILLIAM H. STANTON, former Director and Works Manager of the Philadelphia Quartz Company, Philadelphia, died December 11 at the age of eighty-three. He had been associated with the company since 1889, when he assumed the duties of Superintendent of their Anderson, Ind., works, the oldest PQ Works still producing silicate of soda. He retired from active service in 1919 and pursued as hobbies various earlier interests such as wood turning, travel and family history research. He is survived by his wife.

• NEW SOCMA OFFICE

The office of the Synthetic Organic Chemical Manufacturers Association is now located at 6 East 45th Street, New York 17, N. Y. The new telephone numbers are MUrray Hill 2-8128-9

• HOOKER AWARD

The Hooker Electrochemical Co., Niagara Falls, New York, has been awarded the Army-Navy Production Award for the third time. This was announced in a letter from Under Secretary of War Robert P. Patterson addressed to the men and women employees. This award is symbolized by the addition of a second white star to the "E" flag.

• STEIN, HALL CONFERENCE

Stein, Hall & Company, Inc., manufacturers and distributors of starches, gums, dextrines and specialty products to various industries since 1866, held a Chemists' Conference recently at its main offices, 285 Madison Avenue, New York. About 40 chemists from Stein-Hall's textile, food, paper, adhesive and research laboratories throughout the country participated in the discussions.

The research and development work done in textiles, paper, food and adhesives during the past year was reviewed, and plans were laid for the projects to be concentrated on during the coming year, particularly from the viewpoint of the post-war picture.

The sessions were under the direction of Dr. Alexander Frieden, Technical Director of the Stein-Hall Organizations, and among those present were: G. V. Caesar, Head of the New York Laboratory; F. G. LaPiana, Head of the Providence Textile Laboratory; T. G. Heiser, Head of the Long Island City Laboratory; D. E. Truax, Head of the Charlotte Textile Laboratory; J. V. Bauer, Assistant Technical Director of Stein-Hall Mfg. Co., Chicago; H. F. Gardner, Head of the Chicago Laboratories of Stein-Hall Mfg. Co.; E. F. Glabe, Head of the Food Laboratory of Stein-Hall Mfg. Co., Chicago; H. E. Nelson, Head of the Paper Laboratory of Stein-Hall Mfg. Co., Chicago; E. Percy, Head of Stein-Hall, Ltd., Canada Laboratory.

• 1944 SPRING HOSIERY COLORS

Sundash, a radiant sunblush tone, Honeyglo, a lively tropical tan, and Dawnhaze, a subtle neutral beige, comprise the trio of distinctive colors featured by The Textile Color Card Association in the Confidential Advance Issue of its 1944 Spring Hosiery Card, just released to members.

In accordance with the Association's strict conservation measures adopted at the outbreak of the war, it was stated by Margaret Hayden Rorke, managing director, the hosiery collection is again limited to three colors, all possessing highly individual attributes and each being created to complement a wide selection of important costume tones for spring and summer. Every color has likewise been carefully correlated with the Colors for Shoes, as specified in the War Production Board Conservation Order M-217, as amended October 18, 1943.

The three new colors, it was explained, can be included among the maximum number of colors, in accordance with the War Production Board Hosiery Order L-274. All are adaptable to rayon, as well as cotton and wool hosiery.

• JOINS AMERICAN VISCOSE

John E. VanNest has joined the Textile Research Department of the American Viscose Corporation located at Marcus Hook, Pa., where he will assist Howard E. Shearer in the Industrial Division, it was announced recently.

Mr. VanNest was formerly with the Winthrop Spinning Company, manufacturers of rayon and worsted yarns, and the Wilton Winthrop Mills, woolen manufacturers, holding the position of assistant superintendent at both plants. Prior to this he was associated with the Dana Warp Mills as overseer of rayon carding and spinning.

• CATALOG OF TECHNICAL BOOKS

A new catalog of technical books has just been issued by The Chemical Publishing Co., Inc., 26 Court Street, Brooklyn 2, N. Y. This catalog includes the latest books on chemistry, technology, physics, general science, mathematics, engineering, radio, aviation, foods, formularies, cosmetics, gardening, medicine, metals, technical dictionaries, etc.

This catalog, conforming with the requests of technical and scientific workers and librarians, gives the date of publication of each book as well as a concise description and full table of contents.

• TRADE SALES DIVISION

Effective January 1, 1944, the Trade Sales Division of Interchemical Corporation will become the official outlet for all consumer products developed by Interchemical, its divisions and subsidiaries, with home offices and factory at Paterson, New Jersey.

Two years ago the Setfast Division of Aridye Corporation, an Interchemical subsidiary, was formed to provide an outlet for Aridye products, suitable for the retail market. The first of these products, Setfast Canvas Paint, was so successful that the new Trade Sales Division was formed to provide an outlet for all retail products developed.

SPECTROPHOTOMETRY

(Continued from Page 6)

For instance, if the fastness of Indanthrene Yellow G were being tested when dyed by itself, the colorimetric interpretation of fastness would rate it as poor because it tends to turn olive upon fading. In combination with other colors to make khaki, this dulling of yellow G tends to counteract the fading of the other olive components and the result is improved fastness of the combination. The fact is, therefore, that yellow G is beneficial in the khaki formula. Analytical VS data would make this evident, colorimetric VS data would not.

Fading to tone can be evaluated with the spectrophotometer. Quantitative measurements can be made of the fading of individual components and a mixture which will fade to tone can be predicted quantitatively. Figure 11 shows how three different samples faded. A is the as-is swatch and A prime is the swatch faded. It will be noted that the curve has been raised considerably in the red and in the central part of the spectrum, while in the blue part of the spectrum on the left, it has not been raised very much. This indicates that the yellow component has not faded and therefore sample A is said to fade yellow. For curves B and B′, it will be noted that in the blue end of the spectrum and the red end, there is considerable fading, while in the center of the spectrum there is relatively little fading. This indicates that the red does not fade out and the sample is therefore said to fade red. On the other hand, when C was faded, the faded curve being shown by C prime it will be noted that the faded curve is parallel to the unfaded curve and represents essentially a change with no shade change. Such a change, which is one of strength alone, is usually described by a color-matcher as fading on tone.

Thus fading results may be analyzed from the colorimetric viewpoint which is the equivalent of visual evaluation and gives the extent of color change, or they may be interpreted from the analytical viewpoint to get information on chemical changes, fading to tone, and other aspects.

MAINTENANCE OF STANDARDS

Another application is the maintenance of standards. Figure 12 shows the curve of a yellow wool skein kept in a closed envelope and carefully preserved as a standard. It also shows the curve of another piece of the same material which has been used as a working standard in color matching. Merely by handling the skein it has become duller and dirtier. Periodic checks of standards will call attention to such changes and insure the uniformity of shade over long time periods.

The maintenance of standards is a great problem of the government. In order to procure cloth for uniforms and other purposes from a large number of mills, it is desirable that the same shade swatch be submitted to all suppliers. By colorimetric methods it is possible for a government agency to insure the uniformity of all submitted swatches of standard cloth. VS standardization by the government does not mean that each mill will have to buy a VS and each dyer become a physicist. It merely means that each cloth standard submitted to the dyer will be alike. Neither does it mean that the permitted dyes will be restricted, for colorimetric VS matches may be obtained with different dyes.

DETERMINATION OF TOLERANCES

The VS may be used in the determination of tolerances.[27] It is well known that tolerances vary widely depending upon whether there exists a "buyers" or a "sellers" market.[28] In this connection the importance of light source[29] and viewing conditions[30] is self-evident. Nevertheless this knowledge of what the tolerances are enables one to follow a consistent manufacturing policy. Tolerances may be based either on analytical or colorimetric calculations. In cases where the same dyes are used, it is only necessary to determine the amount of each dye present on the cloth and tolerances may be expressed in terms of percentage variation from the standard. This is the simpler and quicker way but is only applicable to dyeings of the same formulation.

In order not to restrict the dyes used, and yet establish tolerances, as government procurement agencies must do, recourse must be had to colorimetric methods. In one method the number of permissible Judd units difference between the standard and match may be specified. This may not always be entirely satisfactory because the pattern of previously accepted material may be found to be non-spherical. Another method is to define a space in the color solid by ± limits of x, y, and Y. A still simpler method is a limiting percentage on X, Y, Z.

If a government procurement agency specifies tolerances in colorimetric terms this does not mean that every mill must buy a VS and dyers become mathematicians. Normally the difference in the definition of tolerances will not be noticeable in the trade. Most submitted samples will be obviously satisfactory or unsatisfactory by visual tests. Only for a few borderline cases will the VS be called upon to make a decision and here the impartial judgment of the VS will insure a fair deal to all.

The important point is that if a government procurement agency utilizes VS colorimetric methods in maintenance of standards and tolerances the dyer will still have swatches to match, will be as unrestricted as at present in his choice of dyes and mills will not need VS's to operate. The only result as far as the dyer is concerned will be increased uniformity of standard and fair treatment in the classification of questionable samples.

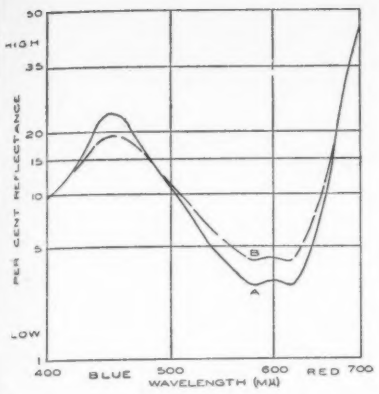

Figure 10
Typical Fading
Anthraquinone Blue RXO (C. I. 1076) 128 hours in Fade-Ometer. Note loss of depth at 500 mu and increase of dullness at 450 mu.

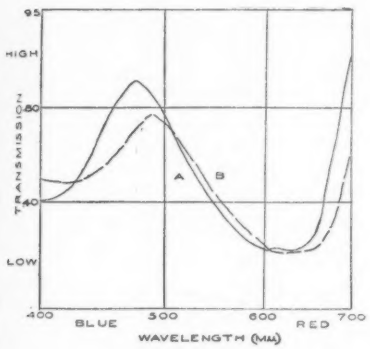

Figure 13
Dye Identification by Curve Shape
A Alizarine Cyanine Green (C. I. 1078)
B Brilliant Benzo Green GL
These have visually similar chemical reactions.

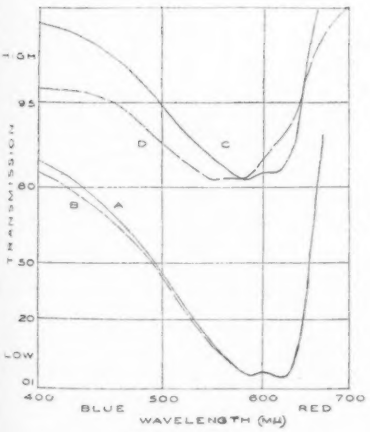

Figure 16
Exhaust Analysis—Detection of Impurity—Acetate Blue
A, B initial solution
C exhaust of A
D exhaust of B

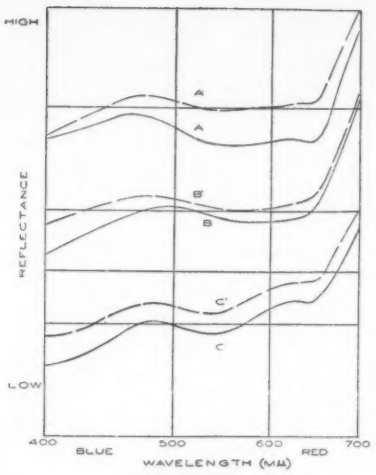

Figure 11
Fading on Tone
A Fades Yellow
B Fades Red
C Fades on tone
Solid lines are original curves
Dotted lines are faded curves

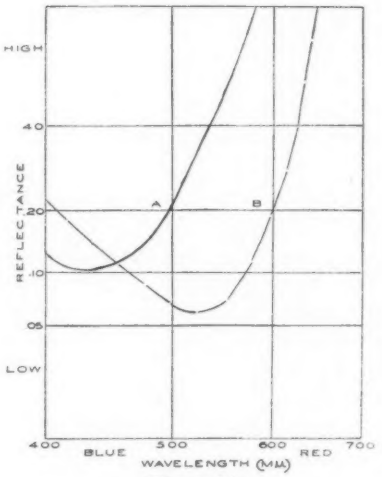

Figure 14
Dye Identification by Chemical Reactions
A As is curve of Brilliant Yellow (C.I. 364) on cotton
B Curve when spotted with caustic

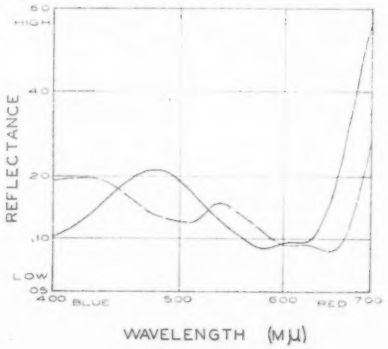

Figure 17
Shade Change in Artificial Light

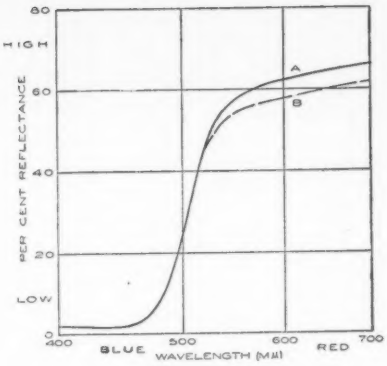

Figure 12
Maintenance of Color Standards
A Original standard
B Standard dirtied by handling in routine color matching

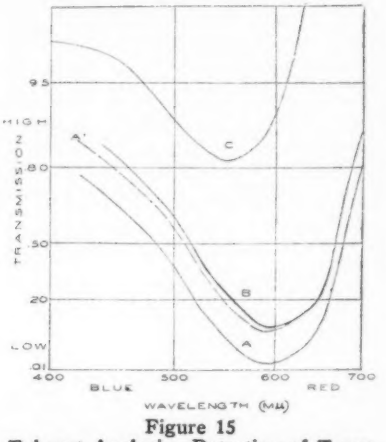

Figure 15
Exhaust Analysis—Detection of Toner
A Alizarin Saphirol B—initial dyebath
A' A displaced for comparison
B Exhaust solution after short time
C Calculated toner—Victoria Fast Violet 2R Ex.

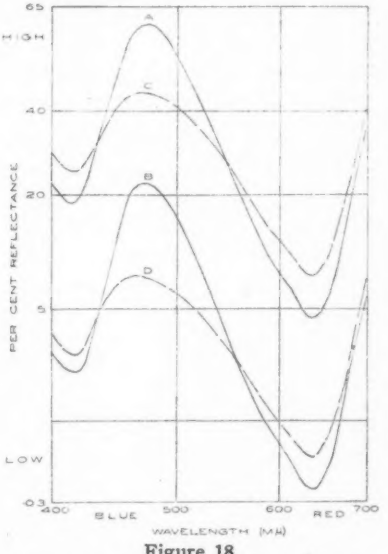

Figure 18
Anomalous Fading of Patent Blue A
A ¼% dyeing—fades lighter visually
B ½% dyeing—fades darker visually
This method of plotting shows little difference in type of fading with concentration.

DYE IDENTIFICATION

In order to offer a better product it is first necessary to find out what one's competitor is doing. Thus one of the first problems of color matching is the identification of dyes in the outside sample in order that one may use the same or better ones. Identification may be made either on the fiber or in solution. The principles of extraction of dye from the fiber[31] and separation by chromatographic analysis[32] are well established in the event that it is desired to work with solution data.

There are two general methods of identification, curve shape and color reactions. Identification by curve shape[19] is illustrated by Figure 13 where two dyes, similar in chemical reaction are easily identified by curve shape differences. Identification by chemical reaction[31,32] is illustrated by Figure 14 where the VS curve before and after spotting with caustic identifies a yellow which is similar to other yellows in curve shape.

OPTIMUM DYEING CONDITIONS

If it is desired to find out what dyeing conditions will give the greatest strength development for any color, a series of dyeings can be made up under different conditions and these can be evaluated by the spectrophotometer. When the VS results are tabulated as in Table III it is very easy to pick out which are the strongest dyeings and these would correspond to the optimum dyeing conditions. It is true that this can be worked out visually but the VS is less laborious because no standard series of type at different strengths need be prepared and because there is no problem of eye fatigue in reading the skeins. It affords greater accuracy because the results can be read to 2% rather than the 5% which is generally accepted as the visual limits. It differentiates between strength and shade because if the log concentration curve is merely displaced vertically by an equal amount at all points, the difference is only one in strength, while if the curve is distorted in any way there is also a shade difference.

EXHAUST ANALYSES

Exhaust data are sometimes very instructive. When a certain sample of Alizarin Saphirol B (C.I. 1054), the initial solution of which is represented by Curve A, of Figure 15 was dyed up, the resulting exhaust was Curve B. When Curves A and B are compared, it is found that they are different.

The color represented by Curve B was subtracted out from the color represented by Curve A prime and the result of this subtraction was Curve C. Curve C, therefore represents a component which exhausted more rapidly than the main body of the dyestuff. A comparison of Curve C with standard curves shows that it agrees exactly with the curve of Victoria Fast Violet 2REx (Pr. 197). It is evident, therefore, that the Violet 2REx was added as a shading toner.

Exhausts also give evidence of chemical identity and the presence of impurities. For instance, in Figure 16 curves A and B represent two samples of an acetate blue

TABLE III

Effect of Varying Dyeing Conditions
Indanthrene Blue BCS
CI 1114

Temperature of Reduction	*Temperature of Dyeing*	*R 4 oz./gal. D 1 oz./gal.*	*R 1 oz./gal. D ½ oz./gal.*	*R 4 oz./gal. D 1 oz./gal. 30% salt*	*R 1 oz./gal. D ½ oz./gal. 30% salt*
180	180	33 m.gr.	47 gr.	53 gr.	39 m.gr.
180	140	48 gr.	65 sl.gr.	57 gr.	65 sl.gr.
180	120	73 v.sl.gr. & v.sl.d.	72 v.sl.gr. & v.sl.d.	72 v.sl.gr. & v.sl.d.	82 v.sl.gr. & v.sl.d.
180	80	63 v.sl.r. & sl.d.	56 v.sl.r. & sl.d.	66 v.sl.r. & sl.d.	69 v.sl.r. & sl.d.
140	140	87 sl.gr.	79 sl.gr.	75 sl.gr.	92 v.sl.gr.
140	120	93 v.sl.gr.	88 v.sl.gr.	92 v.sl.gr.	100 v.sl.gr.
140	80	67 sl.r. & v.sl.d.	63 sl.r. & v.sl.d.	63 sl.r. & v.sl.d.	73 sl.r. & v.sl.d.
120	120	100 sim.	92 sim.	100 std.	100 sim.
120	80	85 sl.r.	70 sl.r.	85 sl.r.	84 sl.r.
80	80	65 sl.r.	57 sl.r.	66 sl.r.	75 sl.r.

Symbols m = much
v = very
sl = slightly
r = red
gr = green
d = dull
sim = similar
std = standard
R = reduction concentration of caustic and hydro
D = dyeing concentration of caustic and hydro

and show that there is a definite chemical difference between the two samples, but one cannot tell whether this difference is due to a difference in the atomic arrangement of the dye molecules or due to some impurity. A comparison of the exhaust of these two colors (C & D) after they were dyed showed a difference due to a dull red non-dyeing component. It is therefore evident that A and B represent in the main chemically identical material, but that B contains in addition to the main dye a non-dyeing reddish impurity.

EXPLANATION OF VISUAL PHENOMENA

The tendency of samples to change in appearance when the illumination is changed from daylight to tungsten can be exactly predicted from the VS curve. Figure 17 shows the VS curves of two grays. The dominant wavelength of A is 3.0 mu less than B under daylight and appears bluer, while under tungsten light, it is 7.5 mu greater than B and appears redder. Thus as the light is changed, the relative shade of A is changed from bluer to redder. Hence the change in hue is exactly predictable.

Patent Blue A (C.I. 714) is noted for its visually anomalous fading. Thus a ½% dyeing on wool exposed

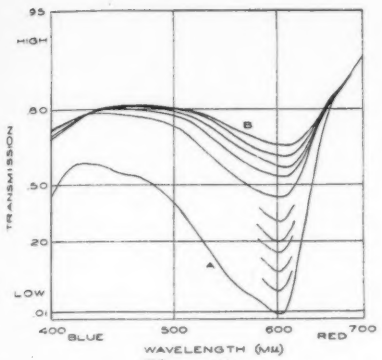

Figure 19
Exhaustion Rate
Caledon Jade Green
A Initial dyebath
B Final dyebath

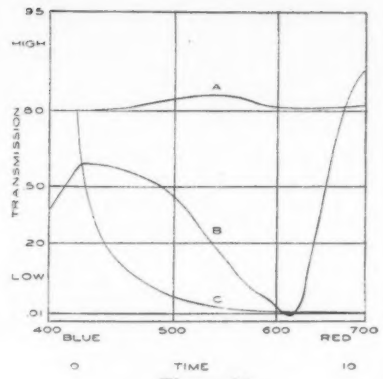

Figure 20
Reduction Rate
Caledon Jade Green
A Initial dyebath
B Final dyebath
Curve C is a time plot of transmission at 610 mu drawn directly by the VS.

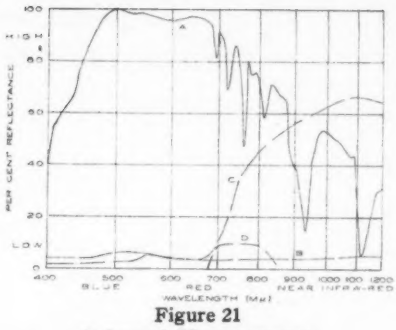

Figure 21
Infra-red Measurements
A Radiation from the sun
D Range of response of infra-red photography
C Heat reflecting dark green
B Heat absorbing dark green

in the Fade-Ometer 8 hours fades lighter. On the other hand a 4% dyeing faded the same time becomes visually darker. Hence the same dye appears to have a different type of fading dependent on the concentration. The analysis of VS data from the colorimetric viewpoint shows this to be true since the overall reflection (Y) of the weak dyeing is increased by fading and is decreased by exposure of the strong dyeing. However, analyzed from the analytical viewpoint when the influence of the color of the wool and the influence of the surface reflectance of the wool are eliminated, and the data are plotted on a log concentration basis, the four curves of Figure 18 are obtained. These curves show that in both cases the fading is about the same, and is the normal type, that is, a loss in depth and a dulling occurring simultaneously. In many cases so called visual fading anomalies are found to be quite normal and entirely predictable from an analytical viewpoint.

The phenomenon of a sample changing shade radically as the concentration is changed, such as Calcocid Ceresine appearing a bluish red at a low concentration (½%) and a yellowish red at a higher concentration (2%) is generally known throughout the trade as dichroism. A better term is dichromatism as dichroism should be reserved for a crystallographic phenomenon of polarized light. Dichromatism results from the fact that if a dye has two regions of high but unequal reflection an increase in concentration will result in the disappearance of the lower one first. Thus in Figure 5a as the bluish red is dyed heavier the VS curve is lowered in the normal, predictable way, but the ratio of blue light (430 mu) to red light (650 mu) has changed from 23% to 10% and the heavy dyeing, with relatively less blue reflected appears yellower.

EXHAUSTION RATES

The idea of following the course of a reaction colorimetrically, proposed as long ago as 1903[34] has recently been improved and applied to the study of chemical reactions and exhaustion of dye from a dyebath.[35,36] Normally only the exhaustion curve is published (see for instance Figure 3 of Royer [36]) as the VS data are only a means to an end. The complete VS curve may be run at stated intervals if the exhaustion is slow, but as in the early part of the exhaustion of Caledon Jade Green (C.I. 1101) in Figure 19 the reaction is so rapid that only a small curve segment may be run. In cases of even greater rapidity the VS may be set to one wavelength and the plot made

TABLE IV
Selected-Weighted Ordinates (Millimicrons)

	Illuminant A			*Illuminant C*		
Weight	*X*	*Y*	*Z*	*X*	*Y*	*Z*
1				415.3		
1				423.5		
1				427.9		
1				431.2		
10				445.5		
1				459.7		
1				462.7		
1				466.0		
1	428.3			469.9		
1	442.1			474.8		
1	452.9	467.4		482.6	446.6	
1	463.6	485.9		520.6	464.0	
1	478.6	495.2	407.7	530.0	473.8	405.7
1	526.8	501.3	415.6	535.3	480.7	413.4
1	535.1	505.8	419.5	539.4	486.1	417.0
10	554.9	520.9	429.0	553.7	504.8	425.9
10	572.5	537.1	438.5	569.7	523.1	434.1
10	584.2	549.7	445.8	582.0	535.8	440.4
10	593.8	560.9	452.4	593.0	546.6	446.2
10	602.6	571.6	458.6	603.9	556.8	451.9
10	611.2	582.3	464.7	615.7	567.1	457.7
10	620.3	593.8	471.4	630.9	578.4	463.9
10	631.3	607.2	479.6		592.1	471.1
10	647.7	625.2	492.1		611.6	481.4
1	667.1	641.7	503.9	643.9	629.2	491.2
1	675.1	646.4	506.8	647.3	634.1	493.8
1	691.5	652.2	510.2	651.4	640.2	496.9
1		660.7	514.3	656.4	648.7	500.7
1		677.8	520.0	663.6	664.6	505.7
1			528.4	677.4		513.1
1			545.4			529.2

Directions for calculating X, Y, Z from a VS curve:
Sum the products of the reflectances at the indicated wavelengths by the weights indicated in column 1. Using these sums proceed as outlined by Hardy[3] as if summations at 100 selected ordinates had been made.

with time as the abscissa. This is illustrated in Figure 20.

INFRA-RED MEASUREMENTS

The infra-red region is of interest to the dyer primarily from the viewpoint of camouflage fabrics[37] heat reflectance,[38] and dye identification.[39] Figure 21 presents the curves of the reflectance of two dark green samples. One of these (B) is infra-red absorbing, the other infra-red reflecting (C). The range of sensitivity of infra-red photography is shown by curve D. Hence this is the region to measure reflectance in camouflage problems. The range of eighty-five per cent of sunlight energy reaching the earth's surface[40] is shown by curve A. Hence this is the region to measure reflectance in heat reflecting problems. The infra-red range is also of use in the identification of some colors, particularly vats, which absorb there.

The above discussion of applications shows that the mill dyer, by taking advantage of the additional information which is available if he uses the VS as a tool to assist him, can be in a better position to solve many of the vexing color problems which are frequently encountered.

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• OPEN FORUM •

58. We are dyeing yarn on perforated tubes, which we call pressure dyeing. Consequently, the dye bath is much more concentrated than its former procedure, namely in skeins.

As a result, on heavy shades we get some sort of a coagulation after dyeing the yarn, and when this yarn is placed on a spool and passed on to the cutting-up trade, the tailors find difficulty in unwinding this thread, as it creates some sort of an electrolitical effect, thus retarding the passage of the thread through the eye of the needle.

Can you help us towards eliminating this problem? Thank you for your cooperation.—**B.S.**

Readers are invited to submit replies to the above question. Payment will be made for those published.

Observations on
SOIL BURIAL PROCEDURES

ELMER C. BERTOLET

Senior Technologist, Engineering Division,
Jefferson Quartermaster Depot

AS a result of extensive work in testing the mildew resistance of jute and osnaburg fabrics used in sand bag construction, the Corps of Engineers definitely abandoned the laboratory accelerated mildew (mold) test in favor of the Soil Burial Test as a measurement of the service life of this material. At the time this decision was made, the only mold test available was the pure culture method employing the mold, *Chaetomium globosum*. In uncontaminated cultures, this organism produces very rapid results but does not distinguish between weather resistant and non-weather resistant finishes. For example, the Chaetomium test indicated that copper resinate was a satisfactory mildewproofing material. The soil burial method proved that copper resinate was entirely unsatisfactory in service and this was substantiated by actual service test.

Under Corps of Engineers Specification No. T-1452A, Processing Fabrics, Threads and Cordage for Mildewproofing, the accelerated mildew infection test is still retained. Chemical tests are likewise employed to determine if the inhibitor is present in amounts required by the specification.

One of the most outstanding mycologists in the United States has expressed the emphatic opinion that the mold cabinet test is worthless. The reasons given for this is that in the mold cabinet the rule of the survival of the fittest prevails, and the "fittest" organism will not necessarily be the one which is most destructive to fabrics. The results in the mold cabinet are, at best, only qualitative. The mold cabinet will show, however, that material will mold.

The methods for conducting the accelerated mildew (mold) test and the soil burial test are given in American Society for Testing Materials Standards, 1942, Tentative Methods of Test for Resistance of Textile Fabrics to Microorganisms, as accepted by the Society at annual meeting in June, 1942.

The accelerated mildew infection test is there recommended as applicable to such fabrics as canvas and duck which are expected to be used in a damp, humid atmosphere.

The soil burial method is given as applicable to sand bag fabrics such as osnaburg which are expected to be in contact with damp soil for long periods of time.

The hazards which Quartermaster Corps fabrics in general must meet usually are much less severe than those met by sand bag fabrics. The greater part of the fabrics procured by the Jeffersonville Depot go into tentage and personal equipment. The fabrics are tightly woven and are of much higher quality than the burlap or osnaburg fabrics used for sand bags and are, in addition, water repellent. The part of the tent nearest the ground is splashed with and comes into contact with the soil. Tents may be dragged over the ground, perhaps muddy, and are often rolled up while wet. All parts of the tent or equipage are at all times inoculated with air-borne microorganisms and are ready to mildew and rot if the proper humidity and temperature condition prevails. Jungle conditions bring about rapid growth.

The method adopted at Jeffersonville in July, 1942, differs from the tentative A.S.T.M. method in several ways. The test specimens used are 6″ x 4″ cut with the longer dimensions parallel to the filling instead of 6″ x 3″ cut warpwise. Twelve instead of twenty specimens of each fabric to be tested are cut, six of the samples to be used for control. The samples are completely buried at a depth of ¼″ for 14 days at 75 plus or minus 5° F. instead of the vertical partially exposed burial at 1″ for 6 weeks at 90 plus or minus 5° F. The reduced time and lowered temperature makes the test less severe and it should more nearly approach the testing requirements of tentage and equipage.

The composted soil burial method was adopted by the Corps of Engineers as the only one available that could be considered to give an accelerated service test. It is admitted that neither for the Corps of Engineers or for the Quartermaster Corps, is the soil burial method without defects. Both the Corps of Engineers and the Quartermaster Corps are interested in light resistance of treatments and the soil burial test gives no information on this point. On the other hand, the soil burial method has so many advantages that, as yet, it has not been improved upon. Some of these are:

(a) It tests the leachability of treatments.

(b) It provides a nutrient medium which is superior to any artificial nutrient that can be produced.

(c) The composted soil provides a buffered environment which cannot be duplicated under artificial conditions, and it is admitted by mycologists that pH is a very important factor in promoting mold growth.

*Presented at Symposium on Fungus Resistance, American Society for Testing Materials, Committee D-13, October 20-22, 1943, New York, N. Y.

(d) The rapidity of the soil burial method compares favorably with that of any laboratory technique that may be advised.

(e) The soil burial method is fool-proof in that it requires no skilled technique in carrying it out.

(f) The results are capable of duplication within fairly narrow limits.

(g) It has the extremely important advantage of eliminating the need for sterilizing treated fabrics. The sterilizing procedure has the effect of ruling out certain of the organic fungicides which are volatile with steam but which may be entirely satisfactory in service.

(h) It provides the aerobic conditions so necessary for the rapid action of cellulose decomposing organisms.

Very recently, Dr. Glenn A. Greathouse, Department of Agriculture, worked out an accelerated laboratory technique that it was thought might duplicate the advantages of the soil burial method. Dr. Greathouse has abandoned the Chaetomium organism in favor of *Metarrhizium*. He has adopted a refined aerobic technique involving the use of a glass fabric substrate, and has tried to duplicate the leaching effect of soil burial by maintaining the circulation of the nutrient solution through the glass fabric substrate. Even though this method would accomplish all of the objectives, it would not improve on the soil burial method.

A soil suspension method for testing mildew resistance of treated fabrics is proposed by Margaret S. Furry and Marian Zametkin, U. S. Department of Agriculture (American Dyestuff Reporter, Vol. 32, No. 19, September, 1943, page 395). In this method, samples to be tested are inoculated by immersion in a soil suspension, and thus are subjected to a mixture of many types of microorganisms which attack cellulose. After inoculation, the samples are incubated under controlled temperature and humidity conditions. It is stated that the tests are completed in a relative short time, and that the procedure is simple and requires neither expensive equipment nor the services of a bacteriologist.

This method has the advantage of using soil bacteria and fungi and not a pure culture like *Chaetomium globosum* or *Metarrhizium sp.* and is thus closer to the inoculation by soil bacteria met with in the field. It has the advantage of using an artificial nutrient which is considered inferior to the nutrient conditions of the soil burial test.

It is assumed that the authors believe the soil suspension method will give superior results over the pure culture methods. If so, it indicates a swing away from the accelerated mildew infection method using pure cultures and a definite trend towards the soil burial test. All that would now be needed for an all-out adoption of the soil burial test would be the replacement of the artificially prepared culture media by the media of the soil by burial. In this way, the soil microorganisms are in their natural environment best suited for propagation and growth.

The agents that would cause deterioration in use of tentage material are excess of moisture enabling microorganisms to grow, and ultra violet radiation. It has been shown consistently that six months' exposure of grey duck to weather will bring about a tensile strength loss of approximately 45 per cent. Just how much of this loss is due to fungi and how much to ultra violet radiation is not known.

Ultra violet radiation may affect the results by affecting both the inhibitor and the material. The cotton fiber is altered chemically so that it reacts strongly with Fehling's solution. The inhibitor might be one that is chemically destroyed and the decomposition products might bring about deterioration of the fiber. The object of mildew test methods is to determine the effectiveness of the mildew inhibitor. The influence of pre-weathering on both soil burial and accelerated mildew tests is believed to be one of acceleration. One bad feature of the soil burial test is that ultra violet radiations are excluded.

The ultra violet lamp has been brought into consideration as a pre-inoculation treatment. A four hour exposure to ultra violet radiation using the Hanovia analytical method quartz lamp, SC-5010, type 7420, before the Metarrhizium test has been suggested as effective in distinguishing between treatments that would hold up on tentage. Accelerated weathering units have also been proposed for this use. It is believed that the Hanovia lamp or any other source of ultra violet radiation is too severe and not representative of the daylight exposure the material would meet in service.

It has been thought that by combining acid leaching, adsorption treatments with Bentonite and other materials, and ultra violet exposures, the soil burial test may be eliminated for sand bag materials except for occasional checks as to the effectiveness of the method for certain new materials that have not been previously tested.

With reference to the accelerated mildew test, it is very difficult to predict what pre-inoculation treatments, if any, should be included to distinguish between good, mediocre, and poor water repellent and light resistant treatments to make such methods predict actual usage for tent materials. Leaching in water for various times and by different methods can be introduced and increase the severity of the test.

At the Jefferson Quartermaster Depot, most materials tested have been given water-repellent treatments. These are exposed to soil burial without previous wetting-out. Samples that are leached before soil burial are buried wet. It is the practice of some laboratories to wet-out all samples in a very weak (0.1 per cent) solution of a wetting agent before burial. Water repellent finishes aid materially in the retention of the mildew inhibitor and so increase mildew resistance.

The sample to be buried is marked with the laboratory test number, using a red crayon pencil, and a straight line is drawn along the filling so that the sample can be properly placed in the tensile strength testing machine after burial. The sample is stapled to the flat side and at the center of a

stick about 4" long, 1" wide and ¼" thick. The test number is marked on the upper half of the stick with an indelible pencil. The lower portion of the stick is pushed into the ground until the sample is flat on the earth. The sample is covered with soil and wet-down. In this way the samples are easily found at the expiration date of the test and the samples can be placed in an orderly manner in rows during burial. After the test, the old numbers are planed off and the sticks used again. Rope and thread are usually identified by means of tags wired to the samples and left exposed during the test.

The over-all test period and the frequency of the observations will depend upon the susceptibility of the test material to the treatment. Leaching, adsorption, and basic exchange in the soil are so severe that it requires some treatment like copper naphthenate to stand up for any length of time in the soil. Soil burial tests will leach out many inhibitors so quickly that no differentiation is possible. This has been recognized and at the Jefferson Quartermaster Depot experimental studies are usually made for 7, 14, 21, 28, 35, and 42 days. The test is discontinued at the end of any period if the fabric falls down. Rope is tested for as long as one year and such tests are in progress. Depot specifications call for 14-day burial with or without leaching in cold, running water for 24 hours. Grey goods which are used as a control and to determine if the soil is active, will be destroyed in approximately seven days.

In placing samples in the soil a rake is used to level the ground. Then soil to the depth of 1" is screened on through a ¼" mesh screen. The soil is leveled and wet-down. The sample is placed in position and soil is screened on to bury the sample to a depth of ¼" and the soil is again wet-down with water. Check tests have shown that more often the soil over the sample is nearer ⅜", seldom ½", and but rarely, if ever, under ¼". About 1" of screened soft soil will wet-down to about ¼". The importance of finely divided soil is obvious if even treatment is expected. The percentage of moisture in the soil during the test approximates 31 per cent. The samples are sprinkled with water twice each day for the duration of the test.

The use of soil that has been sterilized by dry heat or live steam to kill microorganisms (bacteria and fungi), bugs and the like is common greenhouse practice. This treatment makes soil unsuitable for the soil burial test.

The acidity or alkalinity of the soil is a factor in soil burial. The phenolic types of inhibitors will be unduly leached in alkali soil, acid soils will, likewise, affect zinc dimethyldithiocarbamate and sulfides will precipitate mercury inhibitors and make them ineffective. Water used for leaching is variable. The water supply of St. Louis, Missouri, is understood to have a pH of 9 and its use would accelerate leaching of the phenolic types of fungicides, particularly pentachlorphenol.

When the test sample is removed from the soil, it is held under cold, running water and the soil removed by patting or stroking the sample with the hand. The samples are not frictioned upon themselves or on each other but always kept flat and open in the process. The samples are then air-dried, conditioned and tested for tensile strength by the grab method. To pass specifications, the loss in breaking strength of the finished material must not be more than 10 per cent based on the original material. Samples of untreated cotton material buried simultaneously must lose at least 90 per cent of its original strength. This is a check on the efficiency of the burial.

Temperature is important in soil burial and in greenhouses it is not controlled as well as if the burial was conducted in a room capable of being held to constant temperature and humidity conditions. Greenhouses are equipped with heating apparatus but not with cooling equipment so the control would appear to be easier in winter than it is in summer. Thermostatic controls are used and just as soon as the temperature falls below 75° F., steam is automatically passed into the heating coils. It would be easier to control the temperature at 90 plus or minus 5° F. than to control at 75 plus or minus 5° F., the temperature used in Depot tests. Compared to 75° F. the acceleration at 90° F. may be as much as two days in a 14 day soil burial test so temperature is importnt.

For August 1943, temperature readings of soil and room were made twice daily. The soil temperatures averaged A.M. 80.2° F., P.M. 85.2° F., as against 87.2° F. A.M. and 95.8° F., P.M. for the room taken at the same time. The soil temperatures are usually from 5 to 15° F. lower than the air readings due to the cooling effect of evaporation and the lag due to slower heating-up of the soil. The soil temperature ranged between 72 and 94° F., air temperatures between 80 and 107° F. The effect of protracted hot spells is noticed by the closing of the gap between the soil and room temperatures.

Fire, Water and Weather Resistant 12.29 oz. duck containing 0.95% pentachlorphenol was buried at depths of ½", 2", and 4" for 14, 18, 24, 30, 36, 42 days without loss. The same material when exposed to outdoor weathering tests for four months followed by 14 day soil burial test, lost 30% of its tensile strength.

Water repellent No. 4, No. 6, and No. 10 duck, vat dyed olive drab No. 7, containing 0.46%, 0.32% and 0.28% copper respectively was buried in 7 day periods up to 42 days without loss. There was also no loss after 14 day soil burial when samples were leached or were weathered for 30 days. A 180 day weathering test to be followed by 14 day soil burial is continuing.

Water repellent 9.85 duck, vat dyed olive drab No. 4, containing 1% dihydroxydichlordiphenylmethane was buried 7, 14, 21 days both before and after leaching with no loss.

Water repellent 9.85 and 12.29 oz. camouflage printed duck for use in jungle equipment and treated with 0.75% phenyl mercuric triethanolamine lactate on the finished weight of the duck and containing 0.03% mercury gave results as follows:

Burial Test	*9.85 oz. Duck*	*12.29 oz. Duck*
7 Days	6.4%	1.7%
7 Days—Leached	1.8	0.0
14 Days	0.0	19.2
14 Days—Leached	22.7	39.4
21 Days	91.4	75.0
21 Days—Leached	90.4	73.0
28 Days	100.0	73.8
28 Days—Leached	100.0	100.0
14 Days—(Weathered 6 Weeks on Roof)	70.5	20.0

At the concentration used, the results with this inhibitor at the end of 7 days before and after leaching appear to be satisfactory. The results at the end of 14 days have been inconsistent even when the inhibitor has been increased to 1% on the finished weight of the goods. Leaching and weathering adversely affect the results and it is difficult to forecast the service life of this treatment without actual service tests.

Visual examination of samples after burial have shown that, with a good inhibitor, material can come from the soil without any apparent change in strength or appearance. On other occasions depending on inhibitors, the fabric may not lose tensile strength but may show colonies of micro-organisms on the fabric. In a series of tests on the same sample, varying the periods of burial, it has been shown that the strength and appearance of the samples and the extent of mold growth on them is a function of the time that the samples were buried.

The effect of water repellent treatments in conjunction with mildew resistant finishes has already been expressed. Mildew resistant light-weight fabrics, everything else being equal, will not resist soil burial as well as heavier materials. Marquisettes and like materials need the very best inhibitors if they are to resist soil burial. Goods containing size must be thoroughly and evenly desized in order that proper and even application of the inhibitor is obtained or they will fail in burial. Marquisette samples after soil burial have been seen to rot in spots with the surrounding areas remaining quite strong, indicating uneven application of the inhibitor.

Coated fabrics will deteriorate in soil burial due to failure of the base fabric or susceptibility of the plasticizer if the resin itself is not attacked. Acetylation increases markedly the resistance to decomposition in the soil.

It is to be expected that more variation will be found in the tensile strength of buried samples as compared to grey goods. Because of this it is better in any experimental study not to accept results literally but to look upon them as indicative of performance and subject to recheck by duplicate tests.

The soil burial test is an accelerated test that enables quick results instead of waiting as long as one or two years for actual usage tests. The soil burial test must be applied so that the results can be correlated and interpreted into terms of service. Despite variability and other limitations, it provides an accelerated service test that gives results that will check within fairly narrow limits.

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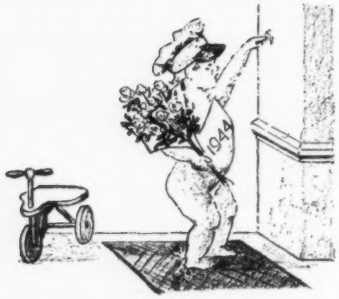

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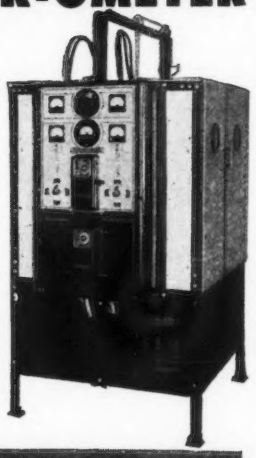

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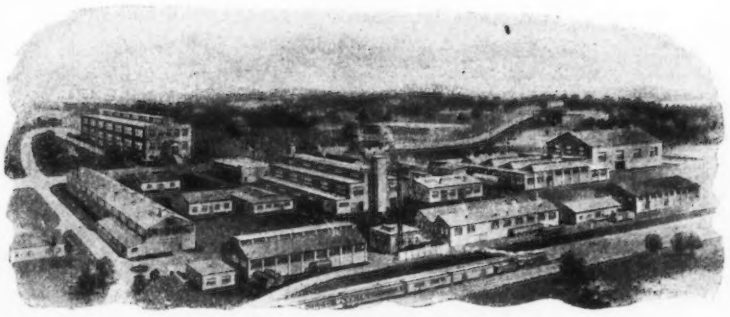

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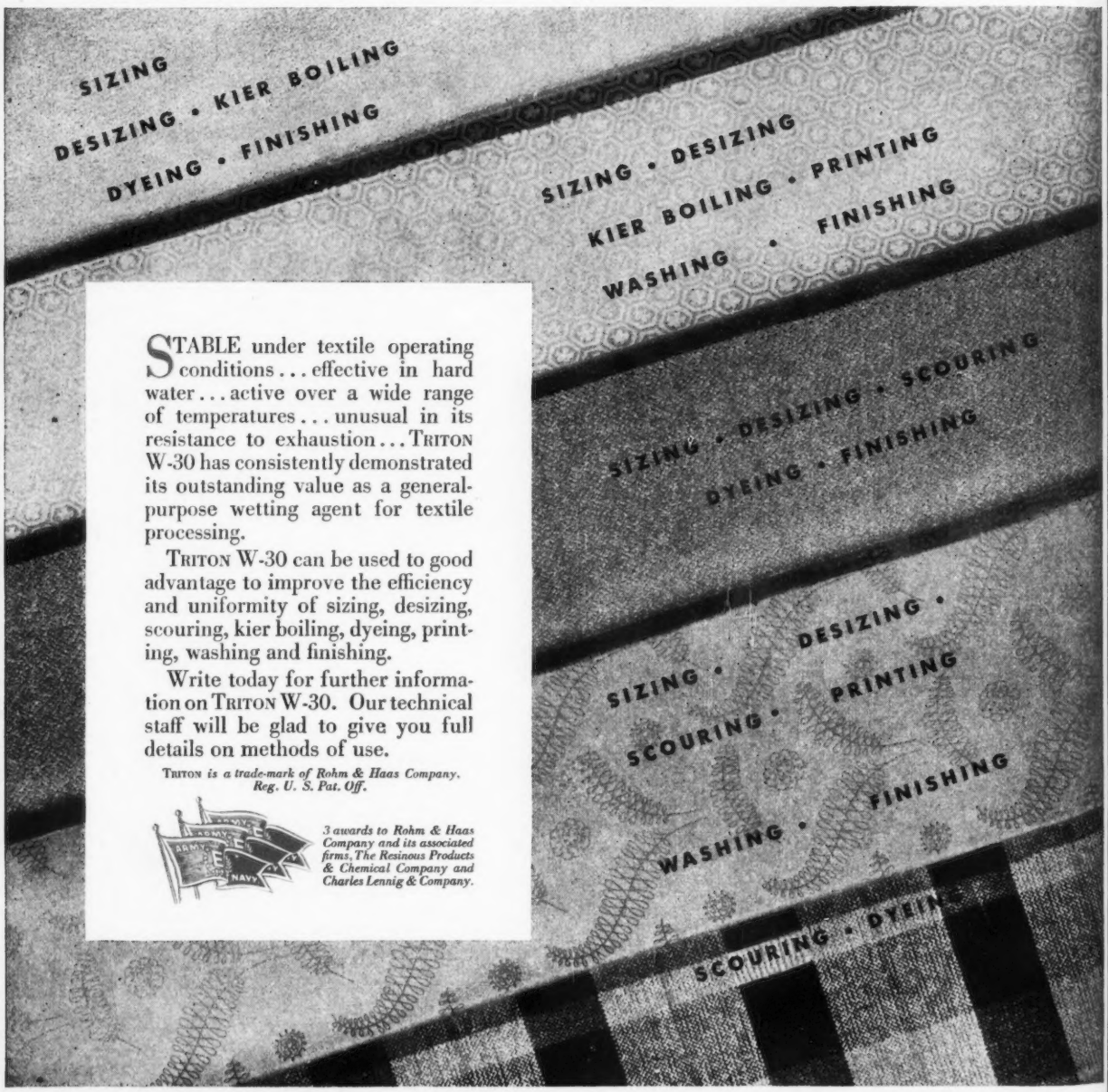

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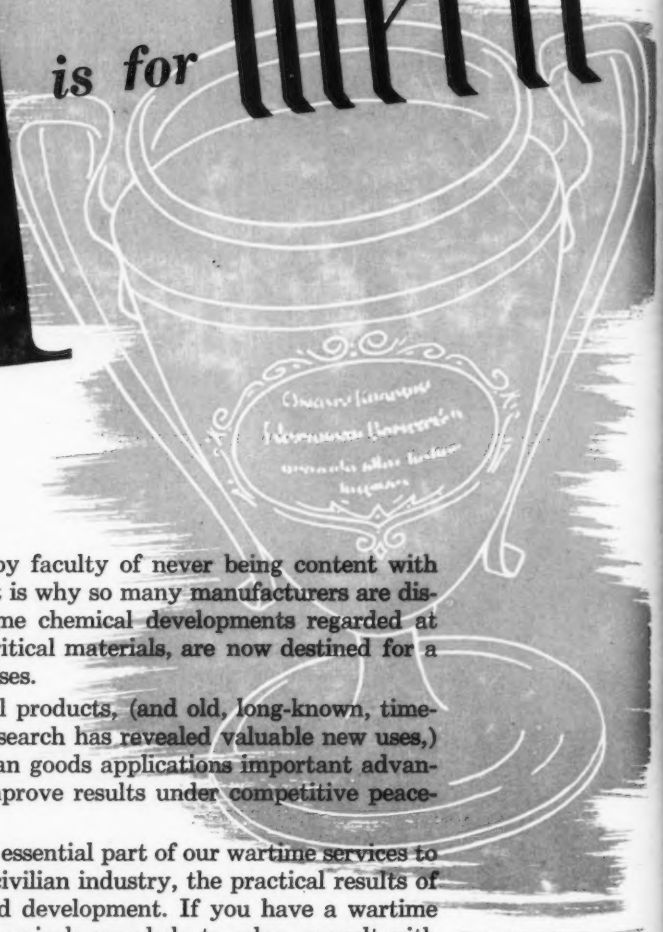

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